Analytical Abstracts

A monthly publication dealing with all branches of analytical chemistry: issued by the Society for Analytical Chemistry

Volume 7
No. 3, Abstracts 848–1232

March, 1960

Published for the Society by
W. HEFFER & SONS LTD., CAMBRIDGE, ENGLAND

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

848. **Rationalisation [standardisation] in chemical analysis.** C. M. Dozinel. *Chim. Anal.*, 1959, **41** (6), 240–246.—Despite the many investigations into optimum conditions for the determination of the majority of elements, most analyses are carried out under conditions and by methods which are known to give inexact results. Two examples, the volumetric standardisation of a soln. of HCl, and the photometric determination of Mn in a standard steel, are cited to show the improvement in both precision and accuracy when a well-proven "rational" method is used by all workers instead of allowing them free choice of procedure. A "rational" procedure should satisfy five criteria—accuracy, reproducibility, simplicity, rapidity and economy in sample, reagents and man-power. The eventual establishment of a European central standardisation body is advocated.

T. R. ANDREW

849. **Symposium on quality control. 10th Anniversary Meeting, American Association of Clinical Chemists, Iowa City, U.S.A.** *Clin. Chem.*, 1959, **5** (4), 300–334.—The following papers were presented. **Statistics in clinical chemistry,** S. M. Free, pp. 300–308. **Use of the control chart in clinical chemistry,** R. J. Henry, pp. 309–319. **System of microchemistry for the hospital laboratory designed for maintenance of a high standard of performance,** D. Seligson, pp. 320–334.

H. F. W. KIRKPATRICK

850. **Determination of molecular weight using thermistors.** J. J. Neumayer (Minnesota Mining and Manfg Co., St. Paul, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (6), 519–523.—An improved application of the principle used by Müller and Stolten (*Anal. Chem.*, 1953, **25**, 1103) substantially reduces the time required for a mol. wt. determination and needs no more than a 5-mg sample. H. N. S.

851. **Application of sodium tetraphenylboron (Kalignost) in chemical analysis.** L. Buzás (Gen. Chem. Inst., Polytech. Univ., Budapest). *Magyar Kém. Lapja*, 1959, **14** (6), 251–257.—A detailed review is given of the applications of sodium tetraphenylboron to the determination of K^+, NH_4^+, organic nitrogen bases and Tl^+. Potassium can be determined by gravimetric, volumetric, conductimetric and amperometric methods, alone and together with NH_4^+; NH_4^+ and organic bases are usually determined by argentimetric titration or potentiometrically. Turbidimetric and radiometric methods have been developed for Tl^+. Most of the processes can also be used on the micro-analytical scale. (110 references.) G. SZABO

852. **Reduction by metals and metal reductors in chemical analysis.** J. Doležal, J. Drahoňovský and J. Zýka (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1959, **53** (5), 481–500.—A review, with 147 references. J. ZÝKA

853. **Spectrophotometric study of acid - base indicators.** Soichiro Musha and Ken'ichi Sugimoto (Fac. of Engng, Univ. of Osaka Prefecture, Sakai). *Japan Analyst*, 1959, **8** (2), 81–89.—The absorption curves of 26 acid - base indicators were examined near the end-point and the results are discussed. The curves are classified into three groups—(*i*) curves showing a max. and a min., the positions of which change with the indicator in the acid and the basic form (*e.g.*, sulphonephthalein dyes); (*ii*) curves in which the max. absorption shifts with change in pH (*e.g.*, azo dyes); (*iii*) curves having an absorption max. the position of which remains unchanged with change in pH, while the extinction coeff. changes (*e.g.*, phthalein dyes). K. SAITO

854. **Triphenylmethane dyes as oxidation - reduction indicators in titrations with cerium(IV) perchlorate.** V. Panduranga Rao and G. Gopala Rao (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **168** (3), 172–177 (in English).—Erioglaucine A (C.I. Acid Blue 9), Erio green B (C.I. Acid Green 16) and Xylene cyanole FF (C.I. Acid Blue 147) can be used as indicators for the determination of Fe^{2+}, U^{4+}, oxalic acid and quinol by ammonium ceric nitrate in 0·5 to 4 *N* $HClO_4$, but cannot be used for V^{4+} or Mo^{5+}, nor for titrations in HNO_3 soln.; As^{3+} can be determined in the presence of 0·1% OsO_4 soln. (1 drop), which catalyses the reaction with Ce^{4+}. The accuracy for Fe^{2+} (0·05 to 2·2), U^{4+} (0·025 to 0·5), oxalate (0·04 to 0·25) and quinol (0·022 to 0·44 millimole) is within ±0·4, 0·2, 0·2 and 0·4%, respectively. J. P. STERN

855. **Iodine trichloride as volumetric reagent. I. Determination of reducing agents.** Balwant Singh and G. P. Kashyap (Panjab Univ., Hoshiarpur). *Indian J. Appl. Chem.*, 1959, **22** (1), 15–18.—Iodine trichloride soln. (0·05 *M*) has been used as a volumetric reagent for the determination of hydrazine sulphate (**I**), As_2O_3 (**II**), $K_4Fe(CN)_6$ (**III**), Na_2SO_3, $SnCl_2$ and antimonyl potassium tartrate (**IV**) at pH 6·5 to 7·5 in soln. buffered with Na acetate, with $CHCl_3$ as indicator. The appearance of a faint violet colour in the $CHCl_3$ indicates the end-point. The reagent has also been used for the potentiometric determination of **I**, **II**, **III**, **IV**, quinol and metol at pH 6·5 to 7·5 in soln. buffered with Na acetate, with bright platinum foil as an oxidation - reduction electrode connected with a S.C.E. through an agar - KCl bridge. O. M. WHITTON

856. **Redox processes in complexometry. III. Systems in which a redox pair, whose potential is modified by EDTA, is employed externally.** G. Asensi Mora. *Quim. e Ind., Bilbao*, 1959, **6** (2), 43–44.—Examples of complexometric titrations are

given in which the redox indicator systems ferri-ferrocyanide-organic base, vanadate-diphenylcarbazone and Fe^{3+}-*o*-dianisidine are used.

L. A. O'NEILL

857. **Flame analysis. A progress report.** A. J. Hegedüs (Forschungsinst. f. die nachrichtentech. Ind., Tungsram, Budapest). *Mikrochim. Acta*, 1959, (5), 735–750 (in German).—A review is presented of flame analysis in all its aspects. (316 references.)

D. F. PHILLIPS

858. **Activation analysis.** D. H. F. Atkins and A. A. Smales (U.K.A.E.R.E., Harwell, Berks., England). *Adv. Inorg. Chem. Radiochem.*, 1959, **1**, 315–345.—A comprehensive review and bibliography is given of the methods employed in applied activation analysis.

K. R. C.

859. **Dielectric constants as characteristic analytical values.** F. Oehme (Inst. zur Entwicklung chem. phys. Analysenmethoden, Weilheim/Obb, Germany). *Angew. Chem.*, 1959, **71** (12), 401–404.—Equipment is described for the measurement of the dielectric constant of liquids, solids and semi-solid or very viscous substances. The dielectric constant (ϵ) is related to the molecular moment (μ) according to the equation—

$$2\epsilon = (4\pi N_L/9kT) \cdot (n + 2)^2 \cdot \mu^2,$$

where N_L is the Loschmidt number, k is the Boltzmann constant, T is the temp. (° K), and n is the refractive index. The possible application of this technique to analytical problems is illustrated by a number of examples, *e.g.*, the autoxidation of turpentine and the effect of the addition of copper and of 1:4-dimethoxybenzene, the analysis of mixtures of *cyclo*hexane and its chlorinated derivatives, the analysis of the 3-component system water-methanol-*iso*propyl alcohol and the analysis of mixtures of rutile and anatase.

T. R. ANDREW

See also Abstract—**1072**, Nitrilotriacetic acid in EDTA.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

860. **Determinations in inorganic analysis by extraction with the use of coloured cations. I. Introduction.** L. Ducret (Éc. Nat. des Ind. Agric. et Aliment., Paris, France). *Anal. Chim. Acta*, 1959, **20** (6), 565–567 (in French).—A brief survey is presented which deals particularly with the uses of the metal complexes of 1:10-phenanthroline and its derivatives and of basic dyes in methods of selective extraction by organic solvents. (51 references.)

H. N. S.

861. **The utilisation of radioactive indicators in the electrolytic separation of metals with a nickel cathode.** R. D. Zhitkov and I. A. Kedrinskiĭ. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 197–203; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,210.—The recovery of Zn in the presence of ions of the alkaline-earth metals was investigated with radioactive indicators and a nickel cathode. It was shown that Zn is deposited quant. by a 90-min. electrolysis from an acetic acid soln. at pH 6·0 to 6·2 with a current density of 0·001 to 0·02 amp. per sq. cm; Sr is only partly deposited under these conditions, apparently as the hydroxide. At pH 4·0 to 4·2 Zn is deposited quant. without co-deposition of Sr.

K. R. C.

862. **Partition between water and chloroform of bivalent-metal chelate complexes of 8-hydroxyquinoline substituted in the 7-position.** F. Umland and K.-U. Meckenstock (Inst. für anorg. Chem., Tech. Hochsch., Hannover). *Angew. Chem.*, 1959, **71** (11), 373.—Complexes of bivalent metals with 8-hydroxyquinoline are rendered readily soluble and stable in organic solvents if the covalency of the metal is raised to six by the addition of two further co-ordinated groups. This can be accomplished by the substitution, in the 7-position of the 8-hydroxyquinoline, of a side-chain $-CHR_2 \cdot NHR_1$; if the groups R_1 and R_2 are large enough, then no extractable complexes are formed with tervalent and quadrivalent metals. An example of such a substituted compound is 7-[α-(*o*-methoxycarbonylanilino)benzyl]-8-hydroxyquinoline (**I**). Copper, Zn, Cd and Hg (10^{-4} *M*) are quant. extracted in one operation by a soln. of **I** in $CHCl_3$ ($< 10^{-3}$ *M*) to give clear soln. which are stable for several hours. Metal complexes with **I** otherwise resemble those with 8-hydroxyquinoline. The pH range for quant. extraction is the same, the absorption max. have approx. the same position ($\simeq$ 380 mμ) and the molar extinction coeff. have the same or higher (Zn, Cd) values. Magnesium ($\simeq 10^{-4}$ *M*) is quant. extracted in one operation by a 0·01 to 0·02 *M* soln. of **I** in $CHCl_3$, but the alkaline-earth metals form no chelate complexes under these conditions. Since KCN will mask most other bivalent metals, **I** is an almost specific reagent for Mg.

J. H. WATON

863. **A standardisation method in the spectrographic analysis of metal alloys.** E. S. Kudelya. *Inzh.-Fiz. Zhur.*, 1959, (1), 46–48; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,596.—To construct a calibration curve, spectra of a single standard are photographed with varied exposures. To obtain comparison lines (*e.g.*, lines of Fe in analysing steel) the exposure is divided into two parts. First photograph the spectrum of the standard and then, on the same portion of the plate, photograph the spectrum of the main component of the alloy (Fe for steel). Photograph the spectra of the samples with full exposure t, the spectra of the standards with exposure t, $\frac{2}{3}t$, $\frac{1}{2}t$ and $\frac{1}{3}t$, and the spectra of Fe with exposure $\frac{1}{2}t$, $\frac{1}{3}t$ and $\frac{2}{3}t$. Correct for contamination of the main component by the elements to be determined. The method may be used for determining V and W in tool steel, Cr, Al and Fe in titanium alloys, and C in ferrous alloys. The calibration curves are rectilinear; the error is not higher than in the normal methods. The procedure cannot be used for elements whose line intensity varies during the exposure.

C. D. KOPKIN

864. **Use of the "cementation" method for concentrating and determining micro amounts of certain metals.** S. L. Dobychin and V. B. Aleskovskiĭ. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 45–48; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,498.—The method described consists in the prolonged shaking of a large vol. ($\simeq$ 1 litre) of the test soln. (pH 3 to 3·5) containing 5 to 100 μg per litre of Cu, Pb, Zn or Cd, 8 g per litre of $MgSO_4$

and 8 g per litre of NaCl, with 0·2 g of metallic magnesium, separating the magnesium, dissolving it in HCl, and determining the metals polarographically in a small vol. of soln. It is shown that this method of concentrating the metals may be used in the determination of micro amounts of these metals in mineral waters, with not specially pure magnesium; in determining lower concn., purer magnesium is required. To determine Cu, Pb, Zn and Cd in the presence of Fe, these metals are first extracted as the diethyldithiocarbamates with CCl_4, the resulting extract is evaporated with HCl, and the metals are determined polarographically with a basal soln. of a mixture of 12·5% KOH and 0·1 *N* aq. NH_3. For concentrating Ni, an excess of Cl^- and acid is necessary; the soln. containing 8 g per litre of NaCl, 8 g per litre of $MgSO_4$ (pH $\simeq$3) and 0·2 g of metallic magnesium is shaken for 5 min., the magnesium is separated and dissolved in 4 ml of conc. HNO_3, and Ni is determined polarographically. The relative error in determining Cu, Pb, Zn, Cd and Ni by the described method is $\gtrsim$20%.

C. D. KOPKIN

865. **Complexometric determination of iodates, chromates and ferricyanides.** S. S. Vil'borg and V. A. Drozdov. *Nauch. Dokl. Vyssh. Shkoly, Khim. i Khim. Tekhnol.*, 1958, (4), 721–723; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,117.—Precipitate iodate from an aq. ethanolic (1:1) or aq. acetone (1:1) soln. as $Pb(IO_3)_2$, dissolve the ppt., after allowing it to settle, in an excess of a standard soln. of EDTA (disodium salt) (**I**) in the presence of ammoniacal buffer soln., and titrate the excess of **I** with $ZnSO_4$ soln. to Acid Chrome dark blue (C.I. Mordant Blue 7) (**II**). This method is suitable for the determination of I_2O_5 after neutralisation with alkali soln. and the addition of a slight excess of HNO_3; the error is $\gtrsim$0·1%. The proposed method for chromates is suitable for their quant. determination in the presence of other oxidants and other cations that interfere in the determination of CrO_4^{2-} by the titration of an excess of Ba^{2+} in soln. The method involves the pptn. of $PbCrO_4$ and the determination of Pb by titration with a soln. of **I**, with **II** as indicator; the error is $\gtrsim$0·5%. Ferricyanides are reduced with KI, the iodine is reduced with thiosulphate, and the soln. is acidified to 2 *N* with respect to H_2SO_4; $(NH_4)_2SO_4$ is added, and $Zn_2Fe(CN)_6$ is pptd. Dissolve the ppt. in **I** soln. and titrate the excess of **I** with a soln. of $MgSO_4$, with Eriochrome black T as indicator. The method may be used for the determination of $Fe(CN)_6^{3-}$ in the presence of cations that react with complexones. The results differ from those by the iodimetric determination by $\gtrsim$0·5%.

C. D. KOPKIN

866. **Quantitative spectrographic determination of thallium, indium, germanium, gallium, tellurium and cadmium in products of lead production.** L. E. Nalmark and I. G. Yudelevich. *Izv. Akad. Nauk KazSSR, Ser. Metallurgii, Obogashcheniya i Ogneuporov*, 1958, (1), 90–98; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,125.—For the determination of In and Tl, mix the samples and standards (1 part) with a buffer mixture (3 parts) consisting of NaCl 45%, PbO 40% and $GdCl_3$ [?] 15%. Place the mixture into a hollow (diam. 4 mm and depth 5 mm) in the carbon electrode. Excite the spectra in an a.c. arc at 8 amp. with an exposure of 50 sec. The slit of the medium quartz spectrograph with a three-lens condenser is illuminated by the central portion of the flame (slit width is 0·01 mm, arc gap 3 mm). In the concn. range 0·001 to 3%, the error is $\gtrsim\pm$10%. The analytical lines used are—In 3256·1 and 2932·6, and Pb 3240·2; Tl 3519·2, 3529·4 and 2918·3, and Pb 3240·2. For the determination of Ga, Ge, Cd and Te, mix the samples with equal amounts of a buffer mixture consisting of carbon powder 86%, NH_4Cl 10%, Bi_2O_3 3%, and SnO_2 2%. Place a three-step neutral filter in front of the spectrograph slit. The analytical lines used are—Cd 3261·1 and Bi 2993·3; Ga 2943·6 and Sn 2913·5; Ge 2651·2, 2709·6 and 2691·3, and Sn 2913·5; Te 2385·8 and Bi 2400·9. The sensitivities of the determinations are—Ga and Ge, 0·0005%; Cd, 0·01%. The error of the analysis is $\simeq$10% (with very low concn., up to 20%). Determine low concn. of Te separately. To increase the sensitivity for Te to 0·002%, increase the current to 11 amp.; illuminate the 0·02-mm slit without lenses, with the arc 165 mm from the slit. Photograph the spectra twice in the same position on the plate. Prepare synthetic standards. Evaporation curves are presented and the basic analytical conditions are given.

C. D. KOPKIN

867. **Studies on the metal complexes of hydroxamic acids. Coloured complexes of iron, vanadium and molybdenum with *iso*nicotinohydroxamic acid and their analytical uses.** R. L. Dutta (Inorg. Chem. Lab., Indian Assoc. for the Cultiv. of Sci., Calcutta). *J. Indian Chem. Soc.*, 1959, **36** (4), 285–286.—The use of *iso*nicotinohydroxamic acid as a colorimetric reagent for Fe, V and Mo is discussed. The reagent behaves similarly to nicotinohydroxamic acid (*cf. Anal. Abstr.*, 1959, **6**, 561). In the determination of Fe, interference by small amounts of Ti, U and Mo may be eliminated by measuring the extinction at 520 to 530 mμ instead of at 440 mμ, but serious interference is caused by V, oxalate and citrate. Vanadium is measured at 510 to 520 mμ in the presence of small amounts of Ti, U and Mo; Fe and citrate cause serious interference. The optimum pH for the development of the Mo complex is 6·5 to 7·8, and measurements may be made at 380 to 390 mμ. Most of the common ions interfere. The compositions of the complexes formed under various conditions are given.

I. JONES

868. **Determination of strong acid mixtures.** R. H. Cundiff and P. C. Markunas (R. J. Reynolds Tobacco Co., Winston-Salem, N.C., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (6), 506–510.—Binary mixtures of H_2SO_4, HNO_3, HCl, H_3PO_4 and $HClO_4$ with each other and with various organic acids are titrated potentiometrically in pyridine with 0·1 *N* tetrabutylammonium hydroxide (*cf. Anal. Abstr.*, 1959, **6**, 439). A mixture of toluene-*p*-sulphonic acid, propionic acid and *p*-benzylphenol is also analysed by this method.

H. N. S.

869. **Spectral determination of low concentrations of deuterium in hydrogen.** A. N. Zaidel' and G. V. Ostrovskaya. *Vestn. Leningr. Univ.*, No. 16, *Ser. Fiz. i Khim.*, 1959, (3), 39–43.—The method of spectral isotopic analysis previously described (Veinberg *et al.*, *Optika i Spektroskopiya*, 1956, **1**, 972) is modified for the determination of D in hydrogen in the range from 0·015% to 10%. The third-order spectrum from a diffraction grating is used, and a filter passing 1% is placed in front of the monochromator slit to intensify the Hβ line. For a D content of 1% the error is $\simeq$2%, rising to 3 to 5% in the range 0·1 to 1% and to 5 to 25% in the range 0·015 to 0·1%. The complete determination occupies 4 min.

W. ROUBO

870. **Moisture determination by elastic scattering of fast neutrons.** W. Kühn (Isotopen-Inst., Wildbad, Germany). *Atompraxis*, 1959, **5** (4–5), 133–137 (in German).—The intensity of the thermal neutrons in the vicinity of a fast-neutron source (*e.g.*, a radium - beryllium source) depends on the H concn., *i.e.*, on the water content, and also on the nature of the substance surrounding the source. Thus, the moisture content of various materials can be determined by measuring the thermal neutrons in the immediate vicinity of the source. Measurements were carried out on anthracite, lignite, sand, wood and raw salts with high water of crystallisation content (carnallite). The influence of density must be taken into consideration with substances whose density is strongly dependent on moisture. NUCL. SCI. ABSTR.

871. **Determination of alkali metals.** M. L. Yashchenko, G. V. Ovchinnikov and L. I. Afanas'eva. Reports of the Fifth Session on the Determination of the Absolute Age of Geological Formations. 1956. Moscow. *Akad. Nauk SSSR*, 1958, 296–307; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,506.—The application of the method of Wells and Stevens (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 236) to the separation and determination of the alkali metals is studied. This method includes the removal of Rb and Cs from K by extraction of the chlorides with ethanol, the separation of Rb and K as chloroplatinates, the removal of Cs from Rb and K with satd. $(NH_4)_2SO_4$ soln., etc. The degree of separation of the alkali metals according to the number of extractions, the adsorption of other ions by ppt. of compounds of one element, etc., is studied by means of ^{42}K, ^{86}Rb and ^{134}Cs as tracers. The advantages are shown of radioactive control of the separations. The procedures studied can be used for the determination of Rb and Cs in mica.
C. D. KOPKIN

872. **Gravimetric estimation of lithium as trilithium phosphate.** K. N. Vishveshwaraiah and C. C. Patel (Dept. of Inorg. and Phys. Chem., Indian Inst. of Sci., Bangalore). *J. Indian Inst. Sci., A*, 1959, **41** (1), 16–22.—The method is a modification of that of Caley and Simmons (*cf. Anal. Abstr.*, 1954, **1**, 238). *Procedure*—To 14 ml of LiCl soln. (5 to 50 mg of Li) add 6 ml of phosphate reagent (100 ml of 2-diethylaminoethanol and 300 ml of water, adjusted to pH 9·5 with H_3PO_4). Heat on a water bath at 65° to 70°, and, 1 hr. after the first appearance of the ppt., add 30 ml of abs. ethanol, with stirring. Heat for a further 2 hr. and filter hot. Wash the ppt. with successive small portions (50 ml in all) of a saturated soln. of Li_3PO_4 in 60% ethanol and finally with 2 ml of hot 80% ethanol. Ignite at 650° to 700° for 30 min. (For 50 to 100 mg of Li use 24 ml of test soln. and 16 ml of reagent; for 100 to 150 mg, use 36 ml of test soln. and 24 ml of reagent; adjust the ethanol concn. to 60% and finally adjust the volume to 100 ml with 60% ethanol.) To the Li found add a solubility correction of 0·35 mg for each 50 ml of final soln. The method is applicable in the presence of Na and/or K, as chlorides, sulphates or phosphates, when the ratios of Li to Na or K, and Li to Na to K are $< 1:5$ and $1:3:3$, respectively, with a reproducibility of $\pm 0{\cdot}02$ mg of Li.
E. C. APLING

873. **Spectrographic determination of lithium and caesium in the flame of thermite flares.** V. B. Aleskovskiĭ, O. N. Setkina, V. A. Kochneva and V. S. Lyadov. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 90–93; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,059.—A thermite mixture of MnO_2 (65%) and metallic Mg (35%) the light of which is free from background, is used for exciting the spectra of Li and Cs. The mixture is easily ignited and burns for a sufficient length of time. The mixture is pressed into 2-g tablets, 10 mm in diam., under a pressure of 5000 kg per sq. cm; a hole of diam. 2 mm and depth 6 mm is made in the tablet, and the sample, as a powder mixed with NaCl, is placed therein. For the preparation of the sample, 1 ml of an aq. soln. of Li and Cs is mixed with 70 mg of NaCl, the water is evaporated off, and the residue is placed in the tablet and covered with a mixture of CuO (65%) and Mg (35%). The tablet is placed on the optical axis of a three-prism glass spectrograph and lit with a match, and the spectrum is photographed. The content of Li and Cs is found from the lines Cs 8521·1 and Li 6708 A, by visual comparison. The presence of Ca does not influence the determination of Cs.
C. D. KOPKIN

874. **Potentiometric micro-titration of potassium.** K. J. Karrman, E. Bladh and P.-O. Gedda (Univ., Lund, Sweden). *Mikrochim. Acta*, 1959, (5), 775–778 (in English).—A sharper end-point is obtained when K tetraphenylboron is titrated potentiometrically in acetone - H_2O mixtures with $AgNO_3$ soln. than when indicators are used. The $AgNO_3$ titre *vs.* K tetraphenylboron in acetone - H_2O mixtures varies with the acetone concn., particularly when this is $< \simeq 40\%$ by vol. D. F. PHILLIPS

875. **Turbidimetric ultra-micro titration of potassium.** K. J. Karrman, E. Bladh and P.-O. Gedda (Univ., Lund, Sweden). *Mikrochim. Acta*, 1959, (5), 779–785 (in English).—The K is pptd. with Na tetraphenylboron and the excess of the reagent is determined by turbidimetric titration with cetyltrimethylammonium bromide. The method is suitable for the determination of < 10 μg of K with an accuracy within 2%. D. F. PHILLIPS

876. **Potentiometric determination of potassium in aqueous solutions.** A. E. Voitsekhovskiĭ and A. F. Strashok. *Trudy Nauch.-Issled. Inst. Osnovoi Khim.*, 1958, **11**, 334–342; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,509.—It is established that the most sensitive electrode pairs for the indirect potentiometric determination of K^+ by using the magnesium salt of dipicrylamine (**I**) are antimony - cadmium and antimony - S.C.E.; K^+ are pptd. with **I**, the excess of which is then titrated potentiometrically with dil. HCl. In determining 0·0709 to 0·1175 g of K^+ the relative error is $\gg 1{\cdot}3\%$. The presence of Na^+ and SO_4^{2-} does not cause interference, but a correction should be made for the content of Fe^{3+} and Al^{3+}. A diagram is given of a semi-automatic apparatus for the potentiometric titration. The determination takes 20 to 25 min. The used soln. of **I** may be regenerated by heating with $MgCO_3$ and treatment of the resulting soln. with HCl; the ppt. of dipicrylamine is washed, dried at 80°, and **I** is re-formed by heating it with $MgCO_3$. C. D. KOPKIN

877. **Gravimetric tetraphenylboron method for potassium in glass and silicates.** H. F. Yankov (Emhart Glass Testing Lab., Bloomfield, Conn., U.S.A.). *Chemist Analyst*, 1959, **48** (2), 38–39.—A sample containing 1 to 10 mg of K is weighed into a platinum crucible and treated with conc. HNO_3;

5 ml of $HClO_4$ and an excess of HF are then added and the soln. is evaporated to dryness. A further portion (2 to 3 ml) of $HClO_4$ is added to glass samples and with felspar, high-alumina samples and lead glass 2 or 3 further additions of $HClO_4$ are made. After heating the residue to incipient fusion, it is cooled and dissolved in water. The soln. is acidified with conc. HCl to give $\simeq$0·5 ml of conc. HCl per 50 ml of soln. and then diluted to 10 to 20 ml. Sodium tetraphenylboron soln. (0·6%) is added, and the determination is completed in the usual manner. Results obtained with various types of glass and silicates are tabulated. The average error with pure KCl was $<$0·07 mg in the range from 1·0 to 10·0 mg of K. A higher precision is attained for commercial glass than with the chloroplatinate method, and with lead glass no preliminary separation of Pb is necessary.

S. M. MARSH

878. **Detection of potassium ion in the presence of ammonium ion.** A. N. Agte. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 182–186; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,507.—In the detection of K^+ with sodium cobaltinitrite, NH_4^+ are removed with powdered metallic magnesium. To the test soln., previously neutralised and tested for the content of Mg^{2+}, add magnesium powder in small portions (0·02 to 0·04 g); heat carefully and after the reaction subsides boil for $\simeq$15 min., periodically adding water to maintain the vol. Test for complete decomposition of NH_4^+ with litmus paper. After the decomposition of NH_4^+, remove the ppt. and test for K^+ in the soln. in the usual manner. The method gives satisfactory results with $\simeq$0·5 mg of K^+ per ml and a ratio of K^+ to NH_4^+ $>$1:100. The presence of hydroxylamine and hydrazine in any concn. does not cause interference in the detection of K^+.

C. D. KOPKIN

879. **Determination of potassium in solutions by its natural radioactivity.** A. A. Tumanov and P. S. Khazanov. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 413–418; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,511.—In the procedure described, a simple liquid counter is used. C. D. KOPKIN

880. **New rapid titrimetric method for copper.** B. K. Sadananda Rao and G. S. Laddha (Univ. of Madras, India). *Anal. Chim. Acta*, 1959, **20** (6), 528–532.—The method gives a positive error of $\simeq$0·5% for 60 to 215-mg quantities of Cu. *Procedure*—To the soln. (100 ml) of Cu^{2+} add 10 to 15 ml of dil. HCl (1:1), 5 drops of 2% aq. $(NH_4)_6Mo_7O_{24}.4H_2O$, 15 to 20 ml of 10% aq. KSCN and a little $NaHCO_3$, and titrate with 0·1 *N* $SnCl_2$ containing 80 ml of conc. HCl per litre. At the end-point, which must be approached slowly, the colour of the soln. changes from pink to red - brown.

H. N. S.

881. **Use of EDTA in chemical phase analysis [of copper and its compounds].** N. A. Filippova and V. A. Korosteleva (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1959, **25** (5), 535–539.—Schemes for phase analysis of mixtures containing metallic copper, Cu_2O, CuO, and sol. salts of Cu^{II} with the use of EDTA as complexing agent are described. G. S. SMITH

882. **Polarographic determination of copper with solid electrodes.** S. M. Gubel'bank and E. N. Lavrinova. *Izv. Vyssh. Uchebn. Zavedenii. Khim. i Khim. Tekhnol.*, 1958, (5), 159–161; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,222.—It is shown that Cu^{2+} are gradually reduced with the formation of two distinct waves of the same height; a platinum cathode and anode are used, 0·5 and 1 mm in diam., 7 mm and 10 mm long, respectively. If the polarography is repeated with the same electrodes without cleaning, a single, composite, easily reproducible curve is obtained that can be used for the polarographic determination of Cu in ores and steel. The supporting electrolyte consists of a mixture of satd. NH_4Cl soln. (200 ml), 25% aq. NH_3 (100 ml), satd. Na_2SO_3 soln. (200 ml) and H_2O (500 ml). *Procedure for ores and steel*—Dissolve the ore (0·5 to 2 g) or steel (1 g) in acid, evaporate the soln. to 2 to 3 ml, add hot satd. NH_4Cl soln. (20 ml) and dilute with the electrolyte to 200 ml. Filter a 100-ml aliquot of the soln. and polarograph. A needle galvanometer can be used when the Cu content is $>$0·5%. The determination is not affected by the presence of W, Mo or $<$5% of V. The results for ores and steel are the same with either platinum or mercury electrodes. K. R. C.

883. **Photometric determination of copper in pyritic residues.** B. V. Mikhal'chuk and Z. A. Sazonova. *Soobshch. o Nauch.-Issled. Rabot. i Novoi Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 76–79; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,062.—Decompose the sample of ground pyritic residue (2 g) by heating for 40 to 45 min. on a sand bath with 50 ml of 10% HCl, cool, make up to 500 ml and filter. Heat 10 ml of the filtrate to boiling, add 10 ml of a freshly prepared satd. ethanolic soln. of tetramethylthiuram disulphide, heat to boiling-point, cool, and extract the resulting ppt. with $CHCl_3$ (2 $\times$ 10 ml). Separate the $CHCl_3$ extract, make it up to 100 ml with 96% ethanol, and measure the extinction in 10-mm cells with a violet filter. The colour is stable for 24 hr. The determination takes 80 min.; the reproducibility is good. C. D. KOPKIN

884. **Complexometric determination of copper and zinc ions when present together.** S. M. Efros, E. S. Bolchinova and Yu. M. Gorfunkel'. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 175–178; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49, 215.—*Procedure*—Acidify the test soln. with 3 *N* H_2SO_4, add 10% aq. NH_3 until a blue colour is obtained and dilute the soln. to 100 ml. Dilute a 10-ml aliquot to 80 ml and determine the total Cu and Zn by titration with EDTA (disodium salt), with murexide as indicator. Pass an equal aliquot of soln. through a cationite column, wash the column with H_2O until the percolate is neutral to methyl orange, then pass 0·25 *N* ammonium oxalate (40 to 60 ml) through the column. Decompose the oxalate complex of Cu thus obtained by heating for 10 min. with 6% H_2O_2 (6 to 7 drops). Add aq. NH_3 to the cold soln. and titrate the Cu with EDTA (disodium salt), with murexide as indicator. Elute the Zn from the column with 3 *N* HCl (50 ml), add NH_4Cl and an excess of aq. NH_3 and titrate with EDTA (disodium salt), with Eriochrome black T as indicator. The error is $>$0·4%; the determination takes 30 min. K. R. C.

885. **Radiometric titration of copper and zinc.** I. M. Korenman, F. R. Sheyanova and N. A. Efremov. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 400–404; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,505.—The copper content of a soln. is determined by radiometric titration with a soln. of

ammonium tetrathiocyanatomercurate (**I**), in some cases by using the radioactive isotope ^{203}Hg, and in others by using the non-isotopic indicator ^{65}Zn or ^{60}Co. Because of its lower solubility, Cu tetrathiocyanatomercurate (**II**) is first pptd., and then the corresponding zinc or cobalt compound. It is also shown that Zn and Co are not co-pptd. with **II**, thus allowing the separate determination of Cu and Zn when present together. To the test soln. add a known amount of labelled zinc or cobalt salt, add the precipitant and measure the radioactivity of the mother liquor. Construct a graph of change of activity *vs.* amount of **I** added. The end-point is found graphically or by calculation. The method may be used for the quant. micro-analysis of brass.

C. D. KOPKIN

886. **Quantitative determination of trace elements by the peak heights on paper chromatograms. I. Determination of copper and nickel ions.** E. S. Boichinova and V. B. Aleskovskiĭ. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 94–100; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,502.—It is shown that the planimetric method is not suitable for the paper-chromatographic determination of Ni and Cu in natural waters. With Ni and Cu as an example, a quant. method is developed for the determination of trace elements in a drop of soln. (0·0015 ml) by the use of the heights of the coloured peaks on precipitation paper-chromatograms. Nickel is chromatographed by the ascending method on paper soaked in an ethanolic soln. of dimethylglyoxime or dithio-oxamide (**I**), and Cu on paper soaked in an aq. soln. of diethyldithiocarbamate or **I**. With movement of the solvent the unprecipitated cation is extracted upwards and reacts with the precipitant to form a coloured peak. The peak height is proportional to the concn. of cation, but depends also on chromatographic conditions. The optimum conditions for determining Cu and Ni are—solvent, a 12 to 17% glycerol soln.; concn. of precipitant, 0·1%. The paper is first treated with 0·013 to 0·05% gelatin soln.; the chromatography takes 60 min. The error of the determination is $\simeq 10$ and $\simeq 50\%$ when determining 2·5 and 0·05 mg of Cu and Ni per ml, respectively.

C. D. KOPKIN

887. **High-frequency titration and estimation of ions. I. Determination of silver.** A. K. Majumdar and A. K. Mukherjee (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **20** (6), 511–514.—The high-frequency method is applied successfully to the titration of Ag^+ with standard soln. of KCl, KBr, KI and NH_4SCN. Bismuthiol II and 2-mercaptobenzothiazole also give good results for quantities of Ag < 15 mg. Bismuthiol I and 2-mercaptobenzimidazole, K_2CrO_4 and KCN do not give satisfactory results, but the reverse titration of KCN with $AgNO_3$ gives a good end-point corresponding to the formation of $K[Ag(CN)_2]$.

H. N. S.

888. **Determination of silver in galena and blende by radioactivation analysis.** D. F. C. Morris and R. A. Killick (Brunel Coll. of Technol., London). *Anal. Chim. Acta*, 1959, **20** (6), 587–594.—Neutron-activation analysis is applied with the use of the radionuclides ^{110m}Ag - ^{110}Ag. The radiochemical separation, after the addition of Ag carrier, is based on the pptn. of Ag as chloride, scavenging with $Fe(OH)_3$, pptn. as sulphide, conversion into oxide and finally into iodate. Radiochemically pure Ag is thus obtained and counted as iodate; the chemical yield is determined gravimetrically. Standards prepared by adding known amounts of Ag to mineral samples are treated similarly. The method is applied to samples of galena containing $\simeq 1\%$ of Ag and to a sample of blende containing 0·0075% of Ag, with results that show a coeff. of variation $< \pm 3\%$. It can be extended to the analysis of samples containing < 0·0001% of Ag.

H. N. S.

889. **Coulometric titration with electrogenerated tin(II). Titration of gold(III) and vanadium(V).** A. J. Bard and J. J. Lingane (Harvard Univ., Cambridge, Mass., U.S.A.). *Anal. Chim. Acta*, 1959, **20** (6), 581–587.—Quantities of Au^{3+} from 1 to 23 mg are titrated in a soln. containing 3 to 4 *M* NaBr, 0·3 *N* HCl and 0·2 *M* $SnCl_4$ with an average error of $\simeq +0·3\%$, the end-point being detected either potentiometrically or amperometrically. The end-point can also be detected spectrophotometrically at 400 mμ, but less precisely. Similar conditions serve for the titration of V^{5+}. With potentiometric end-point detection the deviation is $\simeq \pm 0·8\%$ for 0·5-mg quantities and $\simeq \pm 0·2\%$ for 5·5-mg quantities of V. The spectrophotometric method of end-point detection (at 390 mμ) can be used, but not the amperometric method.

H. N. S.

890. **Use of certain chromotropic acid dyestuffs as reagents for beryllium.** F. S. Frum and G. I. Shilov. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 405–412; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,516.—Eleven azo derivatives of chromotropic acid are studied as reagents for Be. It is shown that the most sensitive is 7-(2-hydroxy-5-sulphophenylazo)chromotropic acid, which can detect Be in soln. at a limiting dilution of 1 in $1·5 \times 10^6$, and the most specific is 7-(3-aminophenylazo)-chromotropic acid, which can detect Be in the presence of salts of Al, Zn, Pb, Cr, Fe, Mn, Ni and Cu. Two procedures are developed for the colorimetric determination of small amounts of Be.

C. D. KOPKIN

891. **Determination of beryllium by the photoneutron method.** G. W. C. Milner and J. W. Edwards (A.E.R.E., Harwell, England). A.E.R.E. Report AERE-R 2965, 1959, 26 pp.—The only nuclide that interacts with γ-rays with energies between 1·665 and 2·227 MeV to give neutrons is ^9Be. Experiments were conducted in which solids and soln. containing Be were irradiated with sources of 100 to 250 mC of ^{124}Sb from which only 0·2% of the γ-rays have an energy > 2·227 MeV, and the neutrons were detected by boron trifluoride counters mounted in paraffin wax surrounding the sample. The chief sources of error were elements with high thermal neutron cross-sections (*e.g.*, B, Cd, Sm and Gd), and if these were present then the presence of good neutron moderators was also important. Between 37·5 and 326 g of a 10% mixture of beryl in quartz could be accommodated in the apparatus, and the limits of detection for such weights were 0·006 and 0·002% of BeO, respectively. The results obtained by the new procedure and a much more lengthy chemical one for the analysis of minerals containing between 0·1% and 12% of BeO were in good agreement.

G. J. HUNTER

892. **Fluorescence reactions of beryllium and aluminium.** I. M. Korenman and I. A. Grishin. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 383–388;

Ref. Zhur., Khim., 1959, (14), Abstr. No. 49,211.—About 150 hydroxyanthraquinone and azo dyes were studied, of which about 50 gave positive fluorescence reactions with Be and Al. To 1 ml of a neutral soln. of a salt of Be or Al was added 0·1 ml of a 0·1% aq. or ethanolic soln. of the dye. Observations were made after boiling the reactants; the fluorescence developed only slowly in the cold. The reactions with Be were 100 to 2000 times less sensitive than with Al. Gallium, Sc, In, Th, Zr, Cr, Zn and Mg gave fluorescence reactions with the dyes studied; salts of Fe^{3+}, Cu, Ca and Ba did not.

K. R. C.

893. **New microchemical test for calcium.** A. Rokosz and J. Bocheńska (Inorg. Chem. Dept., Jagiellonian Univ., Kraków, Poland). *Chem. Anal., Warsaw*, 1958, **3** (1), 33–36.—The soln. containing Ca as $CaCl_2$ is evaporated to dryness on a slide, and exposed to water vapour to give a satd. soln. A thin glass rod moistened with satd. Na acetate soln. is drawn across the drop. Excess of Na acetate must be avoided as it diminishes the sensitivity of the reaction. Characteristic elongated needles of $CaCl_2.Ca(CH_3COO)_2.10H_2O$ are formed, which are examined under a magnification of ×70; ≤35 μg of Ca can be detected in the presence of Mg, Sr, Ba, K, Na and NH_4Cl. Chlorides do not interfere.

H. Dmowska

894. **Volumetric determination of calcium in presence of phosphate.** R. G. Yalman, W. Bruegemann, P. T. Baker and S. M. Garn (Antioch Coll., Yellow Springs, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1230–1233.—An excess (15 to 20%) of a standard soln. (≃0·03 *M*) of EDTA (disodium salt) is added to the sample soln. containing dil. HCl. The pH is raised to ≃13 by the addition of 4 *M* NaOH (5 ml), two drops of calcein soln. are added as indicator, and the excess of EDTA is back-titrated with 0·01 *M* $CaCl_2$ to a yellow - green colour. Finally, EDTA is again added until a pink - brown end-point is reached. Under normal lighting the end-point can be determined with the aid of a spot-plate or by titration in a white opaque polyethylene beaker about one-third full. Small amounts of Mg or Fe do not interfere; it is preferable to remove $Fe(OH)_3$ by centrifuging just before the end-point is reached. The accuracy is satisfactory for samples of bone, teeth and phosphate rock, but is less satisfactory for urine.

W. J. Baker

895. **Analytical chemistry of calcium fluoride.** H. Ballczo and M. Hodos (II Chem. Inst., Univ. of Vienna, Austria). *Z. anal. Chem.*, 1959, **168** (3), 161–165.—The ignition of pure CaF_2, which always contains traces of H_2O, results at 105° in the production of Ca(OH)F and HF and at >400° in further dissociation to CaO and HF. The HF produced may be largely adsorbed on the solid surface, but losses of ≃0·5% at 105° and 2 to 2·5% at 1000° are demonstrated. Of the latter loss, one-half is due to loss of H_2O and one-half to loss of HF. Ignited CaF_2 is hygroscopic (owing to its CaO content) and rapidly bleaches a Zr - alizarin lake (owing to adsorbed F^-). The ignition of CaF_2 - $CaCO_3$ in platinum results in attack on the platinum by F.

J. P. Stern

896. **Spectrographic determination of traces of lithium in metallic calcium by means of a spark discharge.** J. Czakow and E. Weźranowski (Inst. of Nuclear Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (1), 9–12.—Traces of Li (3 to 10 p.p.m.) are determined by converting the sample into chloride, forming it into a tablet with graphite, and measuring the Li line 6707 A *vs.* the background; the tablet is placed on the lower copper electrode, with an upper electrode of spectrally pure C. The high-voltage spark is obtained from a 10-kW generator, and the spectrum is photographed by means of a Hilger monochromator D187 on Ilford special long-range plates. The precision (95% confidence level) is ≃±4%.

H. Dmowska

897. **Flame-photometric determination of small amounts of strontium in the presence of much calcium and barium.** E. Pungor and I. Konkoly-Thege (Inst. anorg. u anal. Chem., Eötvös Univ., Budapest). *Mikrochim. Acta*, 1959, (5), 712–719 (in German).—By using the hydrogen - oxygen flame for investigating the determination of Sr by flame photometry, it was found that ionisation interferences between alkaline-earth metals do not occur. Anomalies were sometimes observed between the amounts of material atomised in unit time and the molecular band emission, and these are explained. A method is given for correcting the error due to this cause.

D. F. Phillips

898. **X-ray powder diffraction patterns of strontium phosphates.** R. C. Ropp, M. A. Aia, C. W. W. Hoffman, T. J. Veleker and R. W. Mooney (Sylvania Electric Products Inc., Touanda, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1163–1166.—Methods are described for the preparation of strontium phosphates, of which 12 have been identified and catalogued according to their X-ray powder diffraction patterns. In their chemical reactions the phosphates of Sr are similar to those of Ca.

K. A. Proctor

899. **Vanadometric determination of barium ion.** S. M. Efros, E. S. Bolchinova and V. F. Chuprik. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 165–168; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,523.—The method is based on the pptn. of Ba^{2+} as $BaMoO_4$, reduction of Mo^{VI} to Mo^V, and titration of the Mo^V with NH_4VO_3 soln. To 10 ml of test soln. (≃100 mg of Ba^{2+}) add 5 to 8 ml of water and aq. NH_3 till just alkaline, warm to between 85° and 90°, add a 2% ammoniacal soln. of ammonium molybdate in excess (≃30%), and heat on the water bath for 30 to 40 min. Filter off the ppt. of $BaMoO_4$, wash it with a satd. soln. of $BaMoO_4$ and dissolve it in 50 ml of 1 to 1·5 *N* HCl. Pass 10 ml of the resulting soln. and 40 ml of 1·5 *N* HCl through a bismuth reductor, add to the filtrate 20 ml of conc. H_2SO_4 and 1 drop of a 0·1% soln. of phenylanthranilic acid, and titrate with 0·1 *N* NH_4VO_3 (≃8 *N* in H_2SO_4) to a red - violet colour. To standardise the soln. of NH_4VO_3, to 10 ml of a soln. of $(NH_4)_2SO_4.FeSO_4.6H_2O$ add 5 ml of conc. H_2SO_4 and one drop of indicator soln., and titrate with the NH_4VO_3 soln. till the colour changes from pale yellow to cherry red. One determination takes ≃2 hr. The error is >2%.

C. D. Kopkin

900. **Separations involving sulphides. III. Separation of molybdenum and tellurium from barium.** M. N. Srivastava (Univ. of Sagar, India). *Anal. Chim. Acta*, 1959, **20** (6), 514–515.—A soln. containing ≃0·05 g of Mo as $(NH_4)_6Mo_7O_{24}$ and 0·1 to 0·18 g of Ba as $BaCl_2$ is treated with a large excess of 2 *N* Na_2S (*cf.* Taimni and Srivastava, *Anal. Abstr.*, 1955, **2**, 3277) added in small quantities with

shaking (to avoid the formation of molybdenum blue), acidified with 2 *N* HCl, heated to boiling and allowed to cool. The pptd. $MoS_3.2H_2O$ is filtered off in a sintered glass crucible (porosity 4), washed successively with water, ethanol and ether, dried in a vacuum desiccator, and weighed. Tellurium (0·05 to 0·12 g) is pptd. similarly from a soln. of K_2TeO_3 and $BaCl_2$ (≡ 0·06 to 0·14 g of Ba), and the collected TeS_2 is dried at 100° to 105°. In each method the Ba^{2+} are determined in the filtrate as $BaSO_4$.

IV. Separation of arsenic, antimony, tellurium and mercury from zirconium. M. N. Srivastava. *Ibid.*, 1959, **20** (6), 516–519.—Solutions containing 0·02 to 0·057 g of As as AsO_4^{3-} and 0·03 to 0·04 g of Zr as $ZrOCl_2$, with sufficient NaOH and Na tartrate to retain ZrO^{2+} in soln., are treated with a large excess of 2 *N* Na_2S and then with sufficient conc. HCl to produce a 6 *N* concn. The soln. is boiled and allowed to cool and the As_2S_3 is filtered off in a sintered glass crucible (porosity 4), washed with *N* HCl, water and ethanol, dried at 100° to 110° and weighed. Zirconium is determined in the filtrate by pptn. with cupferron after adding 10% of H_2SO_4. Antimony, as antimony potassium tartrate, is separated similarly from ZrO^{2+}, but the concn. of HCl is *N* to 1·2 *N*. The Sb is weighed as Sb_2S_3. Tellurium is pptd. as TeS_2 from soln. of K_2TeO_3 and $ZrOCl_2$, and Hg is pptd. as HgS from soln. of $HgCl_2$ and $ZrOCl_2$ by the procedure used for Sb. The method is not suitable for the separation of Se or Sn from Zr. H. N. S.

901. **Photometric determination of zinc with zincon. Application to water containing heavy metals.** J. A. Platte and V. M. Marcy (Hagan Chemicals & Controls, Inc., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1226–1228.—Interference by Cu and other metals in the direct determination of Zn with zincon can be minimised or eliminated by complexing all metals with CN^- and then adding chloral hydrate to free the Zn without destroying the other complexes. *Procedure*—To an approx. neutral aliquot (10 ml, ≃ 10 μg of Zn) add, in the order given and mixing between additions, 1% KCN soln. (1 ml), buffer soln. (pH 9, 5 ml), 0·13% (w/v) zincon soln. (3 ml) and 10% (w/v) aq. chloral hydrate soln. (3 ml). After 2 to 5 min. measure the extinction of the blue Zn - zincon complex at 620 mμ (1-cm cell) against the reagent blank. Beer's law is valid for 5 to 120 μg of Zn in 50 ml of final soln. Cadmium causes high results, and Mn interferes seriously unless Na ascorbate (0·5 g) is added initially to the sample aliquot. For samples of water the error is 4% for 1 p.p.m. and ≃ 10% for 0·1 p.p.m. of Zn, respectively. W. J. Baker

902. **Determination of zinc by the luminescence method with salicylaldehyde semicarbazone.** E. A. Bozhevol'nov. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1958, (22), 65–69; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,227.—The properties of salicylaldehyde semicarbazone (**I**) were studied, with particular reference to its use as an acid - base indicator and as a reagent for the determination of Zn. It was shown that a change in the luminescence of a soln. of **I** from yellow - green through white to blue takes place at pH 6·0 to 8·0. The optimum pH for the determination of Zn was in the range 6·15 to 6·4. In the presence of Zn the blue luminescence was obtained at lower pH values than with pure soln.; Zn was measured by the difference in intensity of colour between a pure soln. of **I** and one containing Zn. The addition of 0·5 ml of a 0·18% ethanolic **I** soln. to 10 ml of test soln. resulted in the max. difference in intensity. With a Zn concn. of ≻ 30 μg per 10 ml a linear relationship was found between the intensity of the luminescence at 470 mμ and the Zn concn.; the intensity decreased in the presence of Cr^{3+} and Co (> 1 μg per ml); Cu, Ni, Fe^{2+} and Fe^{3+} (≃ 1 μg per ml) extinguished the luminescence. The error of the determination (1 to 15 μg of Zn in 5 ml) was < 10%. The method was applied to the determination of Zn (0·002 to 0·004%) in cadmium sulphate and cadmium chloride after the separation of Zn by extraction with *iso*amyl alcohol as the thiocyanate complex. K. R. C.

903. **Influence of metallic lead on the determination of metallic zinc.** B. S. Khristoforov and A. R. Artemenko. *Sb. Nauch. Trud. Vses. Nauch.-Issled. Gorno-Metallurg. Inst. Tsvet. Met.*, 1958, (3), 282–284; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,525.—The determination of metallic zinc in zinc dust is based on the treatment of the sample with a soln. of Fe^{3+} and subsequent titration of Fe^{2+}. If the sample contains other reducing agents the results for Zn are high. The dusts and sublimates of non-ferrous metallurgy often contain metallic lead as well as metallic zinc, and the influence of Pb on the determination of Zn was therefore investigated. After showing, by calculation of the e.m.f. and reaction constants, that the oxidation of Fe^{2+} by Zn and Pb is possible, it is shown that there is a marked difference in the rates of dissolution of pure metallic lead and zinc by the Fe^{3+} soln. No explanation of this difference is given. It is concluded that the influence of Pb on the determination of Zn is negligible and may be ignored in the normal determination of metallic zinc. C. D. Kopkin

904. **Determination of zinc and nickel in an electrolytic bath of black nickel.** S. M. Efros, E. S. Bolchinova and A. K. Kuznetsova. *Trudy Leningr. Tekhnol. Inst. Lensovet.*, 1958, (48), 169–174; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,284.—It is shown that a quant. separation of Zn from Ni is not achieved by the adsorption of these elements on to a cationite resin (H^+ or NH_4^+ form), with subsequent elution of Zn with *N* NaOH and of Ni with 2 *N* HCl. It is suggested that this is due to the formation of nickel zincate, which is insol. in acids and alkalis. An effective separation of Zn and Ni is achieved by the use of a column filled with EDE resin (4 g) that has been washed with 2 *N* HCl. A mixture of the test soln. (4 to 6 ml) and 4 *N* HCl (4 to 6 ml) is passed through and the adsorbed Ni is eluted with 2 *N* HCl (100 ml). The acid eluate is made alkaline with conc. aq. NH_3, murexide is added until a yellow colour is obtained, and the Ni is titrated with 0·1 *N* EDTA (disodium salt); the error is ≻ +0·30%. After the elution of Ni from the column, H_2O (100 ml) and 0·5 *N* HCl are passed through and the eluate containing the Zn is neutralised with aq. NH_3. An ammoniacal buffer soln. (5 ml) is added, followed by Eriochrome black T soln. (a few drops), and the Zn is titrated with *N* EDTA (disodium salt); the error is ≻ 0·38%. K. R. C.

905. **Complexometric determination of zinc, lead and cadmium in brass and bronze.** A. I. Lazarev and V. I. Lazareva (Kuibishev Industrial Inst.). *Zavod. Lab.*, 1959, **25** (5), 542–544.—A tube (16 mm × 250 mm) is filled with a strongly basic anionite, An2F, of grain size 0·5 to 1·0 mm, which has been soaked in water for 4 hr. The column is treated successively with 200 ml of 2 *N* NaOH, 50 ml of

water, and 100 ml of HCl (2 *N* for brass, and *N* for cadmium or lead bronze) at a flow rate of >5 ml per min. For the determination of Zn in brass, the sample (0·25 g) is dissolved in a mixture of 5 ml of dil. HNO_3 (1:1) and 5 ml of dil. HCl (1:1), the soln. is evaporated to between 2 and 3 ml, 15 ml of 6 *N* HCl is added, and the soln. is diluted to 50 ml and passed through the prepared column, after which the column is washed with 150 ml of 2 *N* HCl to remove traces of Cu. The Zn is retained by the column whilst Cu, Fe and Al pass into the percolate, which is rejected. The Zn is then eluted from the column with 150 ml of water, which is passed at a lower rate. For similar further use of the column only washing with 2 *N* HCl is required. The soln. containing the Zn is treated with aq. NH_3 (1:1) until a ppt. appears, then with 10 ml of a buffer soln. containing 5 g of NH_4Cl and 25 ml of 25% aq. NH_3 in 250 ml, followed by 3 drops of 1% Na diethyldithiocarbamate soln. and 6 drops of Acid Chrome blue K indicator, and titrated with 0·1 or 0·05 *N* EDTA (disodium salt). For determining Cd in bronze, the operations are similar, but the washing of the column is carried out with *N* HCl beforehand and also for removal of traces of Cu, the sample soln. also being *N* in HCl, and the Cd, extracted from the column with water, is titrated in the presence of only one drop each of the diethyldithiocarbamate and indicator soln. For determining Pb in bronze containing P, the sample soln. is prepared in 3·5 *N* HCl to prevent pptn. of Pb as $Pb_3(PO_4)_2$. The ppt. of $PbCl_2$ dissolves in the succeeding operations. The acid used in preparing the column and for removing traces of Cu is *N* HCl. The Pb is eluted from the column with 150 ml of water, and the eluate is neutralised by means of aq. NH_3 and HCl, and then mixed with 15 ml of a buffer soln. (prepared by dissolving 10 g of tartaric acid in 90 ml of water, neutralising with 25% aq. NH_3 to methyl orange indicator, and adding 35 ml of 25% aq. NH_3, 5 g of NH_4Cl and 65 ml of water). Titration is carried out in the presence of one drop of the diethyldithiocarbamate soln. and 6 drops of the indicator soln. A better end-point is obtained by addition of excess of EDTA and back-titration with a soln. of Zn in the presence of an ammoniacal buffer soln.

G. S. SMITH

906. **Volumetric determination of mercurous mercury with alkaline permanganate.** I. M. Issa, M. Hamdy and A. El Hadidy (Chem. Dept., Cairo Univ.). *Egypt. J. Chem.*, 1959, **2** (1), 59–64 (in English).—The Hg_2^{2+} are oxidised to HgO with alkaline $KMnO_4$ soln. and the excess of $KMnO_4$ is determined by the addition of $FeSO_4$ and back-titration with $KMnO_4$ soln. *Procedure*—To 0·1 or 0·2 *N* $KMnO_4$ (10 to 20 ml) add sufficient 5 *N* NaOH to make the soln. 0·5 to 1 *N* with respect to the alkali after the addition of the sample. With continuous shaking add the sample soln. containing >2 milli-equiv. of Hg_2^{2+}. After 5 to 15 min. pour the mixture into an excess of standard $(NH_4)_2SO_4.FeSO_4$ soln. containing sufficient H_2SO_4 to give a *N* soln., and titrate the excess of Fe^{2+} with standard $KMnO_4$ soln. The method has a precision of $\simeq 1\%$ for quantities of Hg_2^{2+} up to $\simeq 2$ milli-equiv.

W. T. CARTER

907. **Determination of mercury, copper and zinc by derivative polarography. Application to solutions of uranyl sulphate containing corrosion products.** R. G. Ball, D. L. Manning and O. Menis (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2717, 1959, 26 pp.—Mercury and Cu are determined without previous separation by recording their polarograms, with EDTA, nitrilotriacetic acid (**I**) or Na citrate - ammonium carbonate (**II**) as the supporting electrolyte. In making a choice of medium, the governing factors are the concn. of Fe and the concn. ratios of the components to be determined. In the absence of Fe, EDTA is usually satisfactory. In the presence of small amounts of Fe, **I** can be used to advantage, but in the presence of large amounts of Fe the use of **II** is necessary. From 2 to 20 μg of Cu and Hg per ml can be measured with a coeff. of variation of $\simeq \pm 4\%$. Zinc is separated before the polarographic measurement by complexing it with diethyldithiocarbamate, extracting the complex with $CHCl_3$, and then back-extracting the Zn with dil. HCl. The polarogram is then recorded. The method is applicable to the determination of 2 to 20 μg of Zn per ml with a coeff. of variation of $\pm 2\%$. The effect of foreign substances has been evaluated and their tolerance levels have been established. The greatest interference in the determination of Hg and Cu is due to Fe^{III}; this can be eliminated by the proper selection of supporting electrolyte. Owing to the efficiency of the separation step the polarographic measurement of Zn is relatively free from interference.

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908. **X-ray diffraction determination of boron in boron carbide.** L. M. Osika (Knolls Atomic Power Lab., Schenectady, N.Y., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. KAPL-M-LMO-4, 1959, 8 pp.—By applying the relationships between lattice parameters and composition in the solid soln. range, a method for the determination of B in boron carbide was developed. The purpose was to establish a method of determining B in a boron carbide solid soln. in the presence of excess of carbon or boron. The different unit-cell dimensions sometimes reported for a given solid mixture are usually attributed to the existence of solid soln. If the crystal lattice parameters vary linearly with the composition, a curve can be constructed to determine the composition from lattice parameter measurements. For this purpose four samples of boron carbide of known nominal composition were selected. For each sample, precise lattice parameter measurements and chemical determinations of the boron and carbon contents were obtained to establish the relationship between lattice parameters and boron content.

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909. **Determination of boron-10 in minerals.** G. Allais and H. Curien (Lab. de Minér.-Crist., Fac. des Sci., Paris). *Geochim. et Cosmoch. Acta*, 1959, **17** (1–2), 108–112.—This method is based on the preferential absorption properties of thermal neutrons by ^{10}B; a Ra - α - Be source which emits approx. 3×10^6 neutrons per sec. is used. The rapid neutrons are slowed down by surrounding water, and pass through the boron-containing soln.; In is used as a detector (in the form of a disc 1 cm in diam. and 0·4 mm thick) and absorbs 40% of the thermal neutrons; the activity of the indium is measured by a Geiger - Müller counter. The boron-bearing material is dissolved and 50 ml of the soln. is irradiated for 2 hr. Borax is used as a reference standard. This method has a precision of $\simeq \pm 2\%$ and is simple and rapid, and, assuming that the ratio of ^{10}B to ^{11}B is constant, it can be used satisfactorily for the determination of B. For borosilicates it has the advantage of not requiring the tedious extraction process necessary to eliminate Si.

R. A. HOWIE

910. **Rapid method of determination of traces of boron in graphite and coke by means of spark spectrography.** J. Czakow and T. Steciak (Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (1), 3–8.—Traces of B (0·3 to 10 p.p.m.) in graphite and coke are determined by spectrographic analysis. The ground sample is introduced into the discharge zone between two copper electrodes, the upper one being a "sifter" electrode (*cf.* Feldman and Ellenburg, *Anal. Abstr.*, 1956, **3**, 946). The reproducibility is within ±11·5% for graphite and ±9·5% for coke. If Fe exceeds 0·1%, a spectrograph of high dispersion is used to avoid coincidence of the lines Fe 2497·82 A - B 2497·73 A. Vanadium is used as an internal standard for the line-pair B 2497·73 - V 2520·30. H. Dmowska

911. **Rapid determination of aluminium with high-frequency titration.** E. Pungor and E. E. Zapp (Inst. of Inorg. and Anal. Chem., L. Eötvös Univ., Budapest). *Egypt. J. Chem.*, 1959, **2** (1), 81–88 (in English).—The soln. is titrated with 0·1 *N* $Ba(OH)_2$ at 130 Mc/s. After an initial titration to remove free acidity, sufficient 0·1 *N* $Ba(OH)_2$ is added to convert the aluminium into aluminate, plus a 50% excess, which is then back-titrated with standard acetic acid soln. The end-points are located by graphical methods; CO_2 interferes and must be absent, but SO_4^{2-} can be tolerated if the titration is carried out slowly to allow the $BaSO_4$ to coagulate. Iron causes high results. For quantities of Al between 2 and 14 mg, the precision of the procedure is ≃±3%. W. T. Carter

912. **Indirect EDTA titration of aluminium with lead solutions and 4-(2-pyridylazo)resorcinol (PAR) as indicator.** F. J. Langmyhr and H. Kristiansen (Oslo Univ., Blindern, Norway). *Anal. Chim. Acta*, 1959, **20** (6), 524–527.—In the macro-method, the soln. for analysis contains 25 mg of Al in dil. HCl and has a pH of ≃2. An excess of 0·05 *M* EDTA (disodium salt) is added, the soln. is diluted to 100 ml, aq. NH_3 is added to produce a pH of 6·5 to 7 and the excess of EDTA is titrated with 0·05 *M* $Pb(NO_3)_2$, with 4-(2-pyridylazo)resorcinol as indicator. Soln. containing ≃0·7 mg of Al are titrated with the use of 0·001 *M* soln. When NH_4Cl (10 g) is present the soln. must be boiled before back-titrating, but boiling does not counteract interference due to $NaNO_3$ (10 g). H. N. S.

913. **Determination of gallium in gallium - copper and gallium - magnesium alloys.** E. N. Kunenkova. *Trudy Inst. Metallurg., Akad. Nauk SSSR*, 1958, (3), 289–291; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,530.—The procedures described for gallium - copper alloys are based on the pptn. of Cu with H_2S or $Na_2S_2O_3$, with subsequent pptn. of $Ga(OH)_3$ with aq. NH_3. Alternatively, Ga and part of the Cu are pptd. with aq. NH_3 and the Ga is re-pptd. with pyridine. To determine Ga in gallium - magnesium alloys, re-precipitate $Ga(OH)_3$ with aq. NH_3 in the presence of <10 g of NH_4Cl per 100 ml of soln. C. D. Kopkin

914. **Separation of small amounts of thallium from dilute solutions.** I. M. Korenman and P. A. Ganichev. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 397–399; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,532.—It is shown that, on pptg. Ba or Pb as chromate or sulphate, the Tl present is co-pptd., allowing the almost complete removal of very small amounts of Tl from dil. soln. With an excess of CrO_4^{2-} as precipitant there is substantially complete co-pptn. of Tl (97% with $BaCrO_4$ and 86% with $PbCrO_4$), but an excess of H_2SO_4 leads to a decrease in the co-pptn., which is also somewhat lowered in the presence of colloidal substances. It is established that the co-pptn. is due to adsorption. To remove the co-pptd. Tl from the chromates or sulphates of Ba or Pb, the reaction product of Tl with methyl violet is extracted with benzene. C. D. Kopkin

915. **Microcrystalloscopic reactions for thallic salts.** I. M. Korenman, V. G. Ganina and V. V. Bochkarev. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 389–392; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,533.—Microcrystalloscopic reactions are described for salts of Tl^{3+} with CsCl and hexamine in the presence of KBr, and with thiourea in the presence of $Bi(NO_3)_3$ and HNO_3. The sensitivity and specificity of the reactions and the form and size of the resulting crystals are determined. In selecting the optimum conditions for the reactions, the influence of temperature, concn. of reagents, pH of the medium, and order of mixing the soln. are studied. C. D. Kopkin

916. **Photometric titration of scandium.** J. S. Fritz and D. J. Pietrzyk (Dept. of Chem., Iowa State Coll., Ames, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1157–1159.—A soln. of Sc (0·05 to 3 m*M*) containing 0·05 *M* $Cu(NO_3)_2$ (4 ml) can be titrated photometrically with 0·03 *M* EDTA (disodium salt) at pH 3 and 745 mμ in the presence of as much as a 60-fold molar excess of rare-earth elements. The Sc^{3+} - EDTA complex is colourless, and the extinction remains approx. const. until the end-point (determined graphically), when there is a sharp increase caused by the coloured Cu^{2+} - EDTA complex. The error is ±0·5%. Fluoride, SO_4^{2-}, Bi^{3+}, Th^{4+}, Hf^{4+} and Zr^{4+} interfere, but the sum of Sc and Th can be determined by this method. High concn. of Al, Cu^{2+}, Mg, Fe^{3+} and U^{6+} can be tolerated, under controlled conditions. W. J. Baker

917. **The polarograms of yttrium and lanthanum.** Hiroshi Hamaguchi, Jinzo Hashimoto and Yoshio Narusawa (Fac. of Sci., Tokyo Univ. of Education, Koishikawa). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (1), 43–45.—In 0·1 *N* KCl soln. of pH <3·5, Y gives three polarographic waves: the first is the hydrogen wave ($E_{\frac{1}{2}} = -1{\cdot}37$ V *vs.* the S.C.E.), the second (−1·78 V) is that of the diffusion-controlled 3-electron reduction, the wave height being proportional to the concn. of Y (<1 m*M*), and the third (−1·90 V), the height of which is not proportional to the concn., is that of the 2-electron reduction. In 0·1 *N* tetramethylammonium chloride of pH <3·6, La gives three waves. The first is the hydrogen wave (−1·37 V), the second (−1·89 V) is that of the diffusion-controlled 3-electron reduction, the height being proportional to the concn. of La (<1 m*M*), and the third has a characteristic maximum, the height of which is independent of pH and the concn. of La. K. Saito

918. **Separation of rare-earth elements at amalgam cathodes. III. Separation of samarium from gadolinium.** E. I. Onstott (Los Alamos Sci. Lab., N. Mex). *U.S. Atomic Energy Comm.*, Rep. AECU-4212, [1955], 14 pp.—Samarium is efficiently separated from Gd by electrolysis of the citrate complexes at a lithium amalgam cathode. Under optimum conditions, ≃99% of the Sm is electrolysed while <10% of the Gd is electrolysed, and Gd is deposited

more readily than Dy. Data were obtained which show how the electrolysis is dependent on the cathode potential and the pH of the electrolyte.
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919. **The effect of sulphate ion on the crystal size of precipitated rare-earth-metal oxalates.** A. Glasner, M. Steinberg and E. Levy (Dept. Inorg. and Anal. Chem., Hebrew Univ., Jerusalem, Israel). *Chemist Analyst*, 1959, **48** (2), 37–38.—Large, well-formed crystals of rare-earth-metal oxalates can be obtained by heating a soln. of the oxide in 0·15 *N* H_2SO_4 to 60° and adding *M* oxalic acid, with stirring. After being heated at 60° on a water bath for 2 hr. the soln. is cooled and filtered, and the crystals are washed successively with water, ethanol and diethyl ether. Yields of 82 to 84% are obtained. The effect of SO_4^{2-} is thought to be due to adsorption on the oxalate crystals at points or lines of growth preventing the maturing of crystal nuclei. Since the SO_4^{2-} are in equilibrium with a supersaturated soln., the growth of matured crystallites is slowed down but not inhibited. The crystals contain slightly more water than those pptd. from HNO_3, but no trace of SO_4^{2-}. S. M. MARSH

920. **o-Diketone dioximes and their metal complexes. IX. Photometric determination of the cyanide ion with furil α-dioxime in the presence of nickel.** Kenichiro Yamasaki and Ryojiro Ito (Kita Coll., Osaka Univ., Toyonaka). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (3), 271–273.—Cyanide ions are converted into a nickel complex $Ni(CN)_4^{2-}$ in an ammonium buffer of pH 9 to 11, and the excess of Ni is determined colorimetrically with furil α-dioxime (**I**) in a suspension containing gelatin at pH 4. The colour of the dioxime complex of Ni remains unchanged for 10 min. and the calibration curve is linear for <10 μg of CN^- per ml. No interference results from Ag, Hg, Co, Cu (<1 μg each per ml), Mn (<2 μg), Fe^{III} (<3), Zn (<10), Al (<100), SO_3^{2-} (<30), NO_3^- (<30) or PO_4^{3-} (<800) in the presence of Na tartrate soln. (10%, 2·5 ml). The sample soln. ($\simeq$40 ml) is mixed with ammoniacal Ni soln. (7·5 ml of 0·5% $NiSO_4.7H_2O$ soln., 20 ml of aq. NH_3 and 4 g of NH_4Cl in 100 ml of water) (2 ml), **I** in ethanol (0·5%, 1 ml) and gelatin soln. (0·5%, 1 ml), and diluted to 50 ml, and the extinction is measured at 480 mμ. K. SAITO

921. **Indirect absorptiometric determination of cyanide by means of the mercuric - diphenylcarbazone reaction.** O. Alcides Ohlweiler and J. Oliveira Meditsch (Escola de Engenharia, Porto Alegre, Brazil). *Zhur. Anal. Khim.*, 1959, **14** (3), 303–305.—Hydrocyanic acid evolved from an acid soln. in the outer chamber of a Conway microdiffusion apparatus is absorbed in $HgCl_2$ soln. and the excess of Hg^{2+} not complexed by the cyanide is treated with diphenylcarbazone. Interference by S^{2-} can be overcome by pptn. with $CdNO_3$. *Procedure*—Place a buffer soln. (pH 4) (5 ml) containing 3·00 μg of Hg^{2+} per ml in the inner chamber of the diffusion apparatus, and a soln. (5 ml) containing from 0·30 to 3·5 μg of CN^- in the outer chamber and replace the lid. Add rapidly satd. tartaric acid soln. (1 ml) to the external chamber without removing the lid, mix the soln. by gentle rocking and set aside for 4 hr. at 20°. Add 1% ethanolic diphenylcarbazone soln. (0·1 ml) to the inner chamber and stir; measure the extinction at 550 mμ not more than 5 min. after the appearance of the colour. K. R. C.

922. **Spectrophotometric determination of silica by its molybdovanadate complex.** S. Harel, M. Tamari and C. Klein (Fertilisers & Chemicals Ltd., Haifa, Israel). *Bull. Res. Council, Israel, A*, 1959, **8** (2), 69–74.—The rapid determination of SiO_2 in silicates and fluorosilicates by spectrophotometry is described. The yellow molybdovanadosilicate complex is more sensitive to the acidity of the soln. than is the phosphate complex but, by maintaining a strictly constant acidity, reproducible extinction values can be obtained. For the purpose of the determination, the final acidity is adjusted to 0·2 *N* by adding a reagent soln. of known acidity to a neutralised soln. of the sample. The colour is developed at room temp. ($\leqslant$20°) and the extinction is measured at 390 mμ. The presence of a large excess of F (up to a molar ratio of 15:1) does not affect the results.
J. M. JACOBS

923. **Determination of silicon and phosphorus in reagents without the use of organic solvents.** E. A. Nikitina and M. Z. Partashnikova. *Trudy Vses. Nauch. Inst. Khim. Reaktivov*, 1958, (22), 119–123; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,283.—A method is described for the visual colorimetric determination of Si in carbonates, alkali metals and caustic alkalis; the separation of Si from P and other interfering elements with organic solvents is not necessary. The Si content is determined by the blue colour of the soln. due to the reduction of molybdosilicic acid by $(NH_4)_2SO_4.FeSO_4$. To suppress interference by PO_4^{3-}, the molybdophosphate is destroyed before the reduction by making the soln. *N* in H_2SO_4. The determination of P is based on the visual colorimetric measurement of the yellow colour of molybdovanadophosphoric acid. The presence of $\geqslant$ a 20-fold amount of Si does not cause interference. The methods permit the determination of $\geqslant$0·02 mg of SiO_2 in 80 ml and $\geqslant$0·02 mg of PO_4^{3-} in 50 ml. K. R. C.

924. **Improved sensitivity in the quantitative determination of bismuth, lead, zinc and cadmium in silicon by the sublimation method.** I. B. Borovskiĭ, A. N. Shteinberg and V. V. Bugulova. *Trudy Inst. Metallurgii, Akad. Nauk SSSR*, 1958, (3), 283–288; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,282.—Before making spectrographic determinations of Bi, Pb, Zn and Cd in silicon, a preliminary enrichment is carried out by heating the sample for 4 min. to 1400° in a carbon vessel (7 mm × 12 mm). The volatile impurities are condensed on the end of a water-cooled carbon finger, which is then used as an electrode. K. R. C.

925. **Determination of tin in Zircaloy-2.** U.K.A.E.A. (Res. Group, H.M. Gun Wharf, Chatham, Kent). AERE-AM 58, 1959, 3 pp.—The method is based on standard analytical practice and is described in detail with a drawing of the apparatus by J. Walkden.

926. **Polarographic determination of tin in Zircaloy-2.** T. Bruce and R. W. Ashley (Atomic Energy of Canada Ltd., Chalk River, Ont.). Atomic Energy of Canada Ltd., Rep. CRDC-840, 1959, 8 pp.—The alloy is dissolved in a mixture of HBr and HF, or in a mixture of HBr and HBF_4. Potassium bromide is added as a supporting electrolyte and Triton X-100 as a maximum suppressor. The polarogram is recorded without further treatment of the soln. NUCL. SCI. ABSTR.

927. **Spectrographic determination of tin and vanadium in titanium alloys.** E. S. Kudelya. *Avtomat. Svarka*, 1958, (12), 82–83; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,597.—Excite the spectra in a h.f. discharge, the oscillating and discharge circuits being connected directly, with an oscillating circuit potential of 3500 V, a capacity of 0·01 μF, and a generator feed current of 0·8 amp. Photograph the spectra with a medium spectrograph with slit width 0·025 mm and an upper magnesium electrode. Construct the calibration curves on the co-ordinates ΔS *vs.* log *C* from the lines Sn 3009·1 and Ti 2995·7, and V 2957·5 and Ti 2948·2 A. The error is $< \pm 3{\cdot}5\%$. The determination of Sn and V is carried out simultaneously with that of other elements (*Ref. Zhur., Met.*, 1958, Abstr. No. 7286). C. D. KOPKIN

928. **Complexometric determination of lead by means of the indicators xylenol orange and methyl thymol blue.** A. N. Karolev and M. K. Kolchev (Lead-Zinc Works, Kirjali, Bulgaria). *Zavod. Lab.*, 1959, **25** (5), 546–547.—Sharp end-points in titrations of Pb with EDTA soln. are obtained with xylenol orange at pH 5·4 to 5·9 or methyl thymol blue at pH 5·7 to 6·5. Methods suitable for determining high contents of Pb in concentrates, etc., are described. G. S. SMITH

929. **Polarography of lead in hydroxide and cyanide media.** J. W. Olver and D. N. Hume (M.I.T., Cambridge, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (6), 559–565.—The reduction wave of Pb in cyanide supporting electrolytes is due to the presence of the biplumbite ion and there is no evidence for a lead-cyanide complex in aq. soln. The polarographic characteristics of Pb in alkaline soln. are redetermined in media of constant ionic strength, by using cells without liquid junction potentials. The mercury-mercuric oxide electrode is studied and its use as a reference electrode in polarography is recommended. H. N. S.

930. **Determination of lead in crude copper by alternating current polarography.** Kamihiko Itsuki, Fukunosuke Suzuki and Fumio Sato (Osaka Refinery, Mitsubishi Metal and Mining Co., Shinkawasaki-cho, Kita-ku, Osaka). *Japan Analyst*, 1959, **8** (2), 92–97.—The polarography of Pb (0·1 to 0·5%) in crude copper was examined with a self-balancing a.c. polarograph. The best supporting electrolyte is *M* KCN, containing 1·5 *M* NaOH and 4% of Na_2SO_3. The other conditions are as follows —applied d.c. voltage, 20 mV; rate of flow of Hg, one drop per 3 sec.; temp., 20°; distance between the electrodes, $\simeq$10 mm. The amount of residual HNO_3 affects the result only slightly. There is no interference from the other components of crude copper. *Procedure*—Dissolve the sample (5 g) in HNO_3 (1:1) (40 ml), dilute to 100 ml, and mix a 10-ml aliquot with 7·5 *M* NaOH (10 ml), 5 *M* KCN (10 ml) and Na_2SO_3 soln. (20%) (10 ml). Record the polarogram in the range from 0 to −0·2 V.
K. SAITO

931. **Polarographic determination of lead and zinc in copper mattes.** V. I. Lȳsenko and T. P. Zelenina. *Sb. Nauch. Trud. Vses. Nauch.-Issled. Gorno-Metallurg. Inst. Tsvet. Met.*, 1958, (3), 293–297; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,124.—It is shown that when determining Pb polarographically in copper mattes after decomposing the samples with HCl (1:2) and removing Cu by deposition on iron, low results are obtained because of incomplete extraction of Pb and its partial deposition with Cu. Good results are obtained by decomposing the samples with a mixture of HCl and HNO_3 and separating Pb from Cu by co-pptn. with $Fe(OH)_3$ from an ammoniacal soln. containing NH_4Cl. Heat the sample (0·5 g) for 8 to 10 min. with 10 ml of conc. HCl, add 5 ml of HNO_3 (sp. gr. 1·40), evaporate to dryness, add a further 10 ml of HCl, 5 ml of 10% $FeCl_3$ soln. and 20 ml of water and then aq. NH_3 (sp. gr. 0·91) until a ppt. of $Fe(OH)_3$ is formed and $>$10 ml in excess. Filter off the ppt. of $Fe(OH)_3$ and $Pb(OH)_2$, wash it twice with hot 2% NH_4Cl soln. containing a little aq. NH_3, dissolve it in 50 ml of HCl (1:1) and make up to 100 ml. To 20 ml of the resulting soln. add 0·2 g of metallic iron, and when decolorisation of the soln. is complete add 0·5 ml of a 25% soln. of carpenters' glue, and record the polarogram. Determine Zn, without removing Cu, by polarography with a forced-drop mercury cathode. Dissolve the sample (0·2 g) as described above, evaporate the residue twice with 5 ml of conc. HCl, add 15 ml of 10% HCl, warm, add 40 or 50 ml of basal soln. [100 g of NH_4Cl, 100 ml of aq. NH_3 (sp. gr. 0·91) and 30 ml of a 1% soln. of carpenters' glue in 1 litre] and 10 ml of a satd. soln. of Na_2SO_3, cool, make up to 100 ml with the basal soln., and record the polarogram. The forced-drop method is as accurate as the usual polarographic and potentiometric methods. C. D. KOPKIN

932. **Rational analysis of [lead] products containing elementary sulphur.** B. S. Khristoforov, M. V. Brazhnikova and A. N. Poltaeva. *Sb. Nauch. Trud. Vses. Nauch.-Issled. Gorno-Metallurg. Inst. Tsvet. Met.*, 1958, (3), 279–281; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,538.—The reasons for the unsatisfactory results obtained in the phase analysis of anodic slimes (obtained on the anodic dissolution of mattes in lead production) for lead compounds by the procedure proposed for slags from lead fusion are examined. It is shown that elementary S contained in the slime reacts with the NaOH (used in hot 15% soln. as a solvent for Pb after oxidation), with the formation of Na_2S, which precipitates the oxidised forms of Pb as sulphide. This reaction causes low results for $PbSO_4$ (oxidised) and high results for PbS. The high results obtained in the determination of metallic lead in the slime are explained as being due to the reaction of $AgNO_3$ with the freshly pptd. PbS and the passage of part of the Pb into soln. C. D. KOPKIN

933. **Determination of lead ferrite in slags from lead fusion.** B. S. Khristoforov. *Sb. Nauch. Trud. Vses. Nauch.-Issled. Gorno-Metallurg. Inst. Tsvet. Met.*, 1958, (3), 285–292; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,537.—The usefulness of determining lead ferrite in slags from lead fusion is discussed. From a comparison of the published data on the stability of lead ferrites and the forms in which Fe is found in the slags from lead fusion, it is concluded that the formation of lead ferrites in these slags is impossible, and that the published procedures for determining the various forms of Pb in slags are inapplicable. C. D. KOPKIN

934. **Determination of titanium in beryllium metal.** U.K.A.E.A. (Res. Group, H.M. Gun Wharf, Chatham, Kent). AERE-AM 32, 1959, 3 pp.—The method is based on standard analytical procedure and is described in detail by J. Walkden and K. E. Heathfield.

935. **Colorimetric determination of titanium dioxide in silicates.** J. Debras, A. Debras and I. A. Voinovitch (Lab. de Chim., Soc. Franç. de Ceram., Paris). *Ind. Céram.*, 1959, 117–119.—The hydrogen peroxide and chromotropic acid methods are compared. The colour stability, reproducibility and accuracy of the H_2O_2 method are high, but the sensitivity is poor (<50 μg of Ti should be present in the aliquot), so that values obtained at concn. of $<0{\cdot}2\%$ of TiO_2 can be in error. Measurement of the extinction of the chromotropic acid complex at 470 mμ and pH 2·6 is more rapid, accurate and sensitive for these low concn., whilst the standard deviation (15 determinations) is much less (0·01 to 0·02 as compared with 0·04 to 0·11 for the H_2O_2 method). W. J. BAKER

936. **Platinum-flux technique for determining oxygen in titanium.** W. R. Hansen, M. W. Mallett and M. J. Trzeciak (Battelle Memorial Inst., Columbus, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1237–1241.—Because the reproducibility of the platinum-bath method is unsatisfactory, a simpler modified procedure was developed for determining O in titanium and its alloys. The sample ($\simeq 0{\cdot}25$ g) is wrapped in <2 g of platinum and dropped into the de-gassed graphite crucible in a vacuum-furnace at 1850°. The gas evolved during 20 to 30 min. is collected, and corrections are made for the furnace blank and the O in the platinum. Recoveries of O are 92 to 93% for unalloyed and alloyed samples, and the reproducibility is satisfactory. The dry-crucible method (*cf.* Walter, *Brit. Abstr. C*, 1950, 319) gives similar recoveries and reproducibility. The platinum-flux procedure obviates some of the disadvantages of the dry-crucible, iron-bath and platinum-bath methods, and one analysis can be made in $\simeq 1$ hr. W. J. BAKER

937. **Determination of hydrogen as an impurity in zirconium and Zircaloy.** P. Elbling, G. W. Goward and R. K. McGeary (Westinghouse Electric Corp. Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-183(Rev. 3), 1958, 6 pp.—Hydrogen is extracted from the metal or alloy by induction heating at 1100° to 1200° in a molybdenum or graphite crucible *in vacuo*. The method covers the determination of H in zirconium and Zircaloy-2 in the range of 1 to 3000 p.p.m. on a 0·5-g sample. NUCL. SCI. ABSTR.

938. **Determination of nitrogen in zirconium and zirconium-base alloys.** G. J. Harter, A. W. Perrine and J. F. Rodgers (Westinghouse Electric Corp. Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-CTA(GLA)-170(Rev. 2), 1958, 6 pp.—The sample is dissolved in HCl - HF, and the resulting soln. is made alkaline with conc. NaOH soln. The N is steam-distilled off as NH_3, which is determined spectrophotometrically. The method covers the determination of N in zirconium, Zircaloy-2 and Zircaloy-3 in the range from 5 to 120 p.p.m. on a 1-g sample. NUCL. SCI. ABSTR.

939. **Determination of aluminium as an impurity in zirconium and Zircaloy.** R. M. Burd, G. W. Goward, M. D. Hartman, M. A. McCracken and B. B. Wilson (Westinghouse Electric Corp. Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-C(AR)-143(Rev. 1), 1957, 7 pp.—The bulk of the zirconium is separated from Al as fluorozirconate by ion exchange. Aluminium is eluted with HCl - HF and extracted as the Al - 8-hydroxyquinoline complex with $CHCl_3$; it is then measured fluorimetrically. NUCL. SCI. ABSTR.

940. **Determination of nickel in zirconium and in Zircaloy-2.** U.K.A.E.A. (Res. Group, H.M. Gun Wharf, Chatham, Kent). AERE-AM 50, 1959, 3 pp.—The method is based on standard practice and is described in detail by J. Walkden.

941. **The thoron colorimetric method for thorium determination: effect of some common ions, and methods for overcoming interference.** J. A. F. Bouvier and R. J. Guest. Rep., Dept. of Mines and Tech. Surveys, Mines Branch, Canada, 1958, 17 pp. —A comparison was made of two reducing agents, ascorbic acid and hydroxyammonium chloride, for overcoming the interference of Fe and Ce. Low results for Th were caused by Mo in the presence of ascorbic acid, but this effect was not found when hydroxyammonium chloride was present, or in the absence of a reducing agent. Titanium caused low results for Th under any of the conditions tried, and a very low tolerance limit must be set for this ion. Zirconium caused high results for Th under normal conditions, but its effect varied with the form in which it was added. By using *meso*tartaric acid as a masking agent, $\simeq 5$ mg of Zr could be tolerated in the final 25-ml aliquot taken for the colorimetric measurement. In the absence of Zr, *meso*tartaric acid is not required and a final aliquot of 250 ml is used. NUCL. SCI. ABSTR.

942. **Spectrochemical determination of thorium by d.c. arc.** M. D. Marinković. *Bull. Inst. Nucl. Sci. "Boris Kidrich," Belgrade*, 1959, **9**, 215–217.—A modification of the method of Dutra and Murata for the direct determination of thorium in minerals is proposed. Errors reducing the accuracy of the spectrochemical analysis are discussed. By using CuO as a buffer, the background in the spectra was reduced, leading to more accurate results at lower concn. The precision for samples at different concn. was $\simeq 4\%$ in terms of standard deviation. The max. error was $\simeq 8\%$, in good agreement with the data of Dutra and Murata. NUCL. SCI. ABSTR.

943. **Separation of thorium from rare earths, zirconium and other elements by cation-exchange chromatography.** F. W. E. Strelow (Nat. Chem. Res. Lab., C.S.I.R., Pretoria, S. Africa). *Anal. Chem.*, 1959, **31** (7), 1201–1203.—The sample soln. is passed through a column (20 to 25 cm × 1·5 cm) of AG50W-X12 resin (200 to 400 mesh) at a rate of $0{\cdot}8 \pm 0{\cdot}1$ ml per min. Zirconium and rare-earth elements are eluted from the column with 4 *N* HCl; the retained Th is then determined by igniting the resin at 1250°, and weighing the Th as ThO_2 (a correction is made for the ash of the resin). The separation from Zr and Ce^{3+} is highly efficient, and up to 350 mg of Th in the presence of an equal amount of the other elements can be handled by one column. The method is useful for determining Th in monazites for geochronological work. W. J. BAKER

944. **Direct method for determining nitric acid in sulphuric acid and mixtures.** M. G. Ivanova. *Soobshch. o Nauch.-Issled. Rabot. i Novoĭ Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 89–96; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,120.—To determine HNO_3 in sulphuric acid, cool 200 g of the acid to 0° and titrate with a soln. of $FeSO_4.7H_2O$ (176·5 or 50 g per litre) to a pale-pink

colour. Determine HNO_3 in a mixture analogously. The determination takes 10 to 12 min.; the relative error is 1 to 2%. The method may also be used for detecting $<0{\cdot}0001\%$ of HNO_3 in sulphuric acid.
C. D. KOPKIN

945. **Colour reaction of nitrites.** I. M. Korenman and V. I. Yunina. *Trudy Khim. i Khim. Tekhnol.*, 1958, (1), 144–147; *Ref. Zhur., Khim.*, 1959, (6), Abstr. No. 19,129.—The effect of a large number of dyes containing a primary amino group on NO_2^- in acid soln. was studied. It is shown that many of these dyes can be used as sensitive reagents for the detection of NO_2^- at a limiting dilution of 1 in 3×10^6. The spectrographic characteristics of acid soln. of several dyes and of the products of their reaction with NO_2^- are given. With low concn. of NO_2^- the diazotisation reaction is almost complete 10 min. after introduction of the reagent, except with neutral red, which requires 30 min. A 0·05% aq. soln. of the appropriate dye (0·25 ml) and *N* HCl (0·5 ml) are added to the test soln. (3 ml) and the extinction of the coloured soln. is measured in 1-cm cells at the wavelength of max. extinction of the reaction product; the error of the determination is $>5\%$.
K. R. C.

946. **Estimation of phosphorus as quinoline molybdophosphate.** A. N. Dutta and N. Gupta (Indian Bur. of Mines, Delhi). *Indian J. Appl. Chem.*, 1959, **22** (1), 9–14.—The determination of P in various ores and minerals, in the presence of soluble silica and HCl, by Wilson's method (*cf. Analyst*, 1954, **79**, 535) with modifications, has been examined. In standard phosphate soln. containing 0·5 to 2 mg of P, in the presence of 25 to 500 mg of soluble silica, P was determined by pptn. as quinoline molybdophosphate, followed by titration as in the standard molybdate method, with thymol blue - phenolphthalein as indicator. The effects of varied amounts of citric acid, Ti and V have been studied. Applications of the methods are discussed.
O. M. WHITTON

947. **Determination of orthophosphates, based on the precipitation of bismuth phosphate in acid medium.** I. K. Krotova and M. L. Chepelevetskiĭ. *Soobshch. o Nauch.-Issled. Rabot. i Novoĭ Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 58–67; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,104.—The solubility product of $BiPO_4$ is found to be $7{\cdot}24 \times 10^{-23}$. A method developed for the determination of phosphates is based on the complexometric titration of Bi, with KI, catechol violet or pyrogallol red as indicator. Decompose 2·5 g of the phosphate by heating with 50 ml of HNO_3 (sp. gr. 1·355) make up to 250 ml and filter. To 25 ml of filtrate add an equal vol. of 10 *N* aq. NH_3 (to give a soln. *N* with respect to HNO_3), slowly add 3 ml of freshly prepared 0·1 *N* ascorbic acid, 5 ml of 8% $Ba(NO_3)_2$ soln., 8 ml of 50% $Al(NO_3)_3$ soln., 4 g of $NaNO_3$, 25 ml of 0·1 *M* $Bi(NO_3)_3$ and 10 *N* aq. NH_3 to give a 0·4 *N* acid soln., make up to 100 ml and filter. Dilute 20 ml of the filtrate, adjust the soln. to a pH between 1·5 and 2 (universal indicator paper), and titrate with EDTA (disodium salt) soln. in the presence of 1 ml of 0·5% KI soln. to a pale-yellow colour, then add a further 9 ml of the KI soln. and titrate till colourless. The method gives satisfactory results.
C. D. KOPKIN

948. **Determination of micro amounts of arsenic with a bivalent chromium salt.** P. P. Shat'ko, N. T. Vasina, V. I. Podol'skaya, L. A. Malkina and T. F. Ponamareva (Lugansk State Med. Inst., USSR). *Zhur. Anal. Khim.*, 1959, **14** (3), 358–359.—*Procedure*—Evaporate the test soln. to 10 to 20 ml on a water bath, add HCl (sp. gr. 1·19) (3 to 5 ml) and $CuSO_4$ soln. (2 ml, $\simeq 0{\cdot}01$ g of Cu) and pass a current of CO_2 through the soln.; add $\simeq 0{\cdot}5$ *N* $CrCl_2$ (7 to 10 ml), heat to boiling in a current of CO_2 and boil for a further 2 to 3 min. Wash the ppt. with 2% aq. NH_4Cl soln. and oxidise the soln. with 40% $(NH_4)_2SO_4.Fe_2(SO_4)_3$ soln. (10 ml) to dissolve the Cu. Filter, wash the As remaining on the filter with HCl (1 : 4) and then with 2% NH_4Cl soln. until the pH is 3·75 and determine the As iodimetrically. The method is sensitive to 0·02 mg in 200 ml, and was tested on standard samples of bronze and brass.
K. R. C.

949. **Photometric determination of niobium in carbide mixtures.** R. M. Veĭtsman (All-Union Sci. Res. Inst. of Hard Alloys). *Zavod. Lab.*, 1959, **25** (5), 552–554.—A method based on the formation of coloured heteropoly acid complexes of P, Mo and Nb is described.
G. S. SMITH

950. **Determination of tantalum and niobium in ores and ferro-alloys.** H. Wirtz and H. Rothmann. *Z. Erzbergb. Metallhüttenw.*, 1958, **11**, 465–470.—In addition to 50 to 60% of Nb_2O_5 and 5% of Ta_2O_5, the ores contain TiO_2, SnO_2, MnO, CaO, SiO_2, Al_2O_3 and 19% of FeO. The ores are extracted with alkali and this is followed by hydrolysis of the Ta, Nb, Ti and Sn hydrated oxides (containing WO_3 and P_2O_5). After extraction with $K_2S_2O_7$, Ti and Sn are removed with HCl and H_2O_2. The ppt. contains the bulk of the Ta_2O_5 and Nb_2O_5, while the filtrate contains Ti, Sn and small amounts of Ta_2O_5 and Nb_2O_5. The ppt. is repeatedly treated with HCl - H_2O_2. The filtrate is treated with HCl - H_2O_2 to precipitate the remaining Ta_2O_5 and Nb_2O_5, which is filtered off. Tin is determined as SnS in the filtrate and is extracted with $(NH_4)_2S$, which leaves behind a ppt. of the remaining tantalum and niobium oxides. The combined ppt. are purified from Ti and Fe by extraction with $K_2S_2O_7$ and hydrolysis with HCl - H_2O_2. The wt. of the ppt. gives Ta_2O_5 plus Nb_2O_5 (plus WO_3 and P_2O_5). The WO_3 is determined photometrically and the P_2O_5 by pptn. with quinoline molybdate.
CHEM. ABSTR.

951. **Semi-micro determination of combined tantalum and niobium with selenous acid.** F. S. Grimaldi and M. Schnepfe (Geol. Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1270–1272.—The procedure of Alimarin and Stepanyuk for the pptn. of Nb and Ta with 10% selenous acid (*cf. Anal. Abstr.*, 1957, **4**, 1513) has been modified by adding H_2O_2 during dissolution of the cooled melt and carrying out the pptn. at 100° from a vol. of 150 ml containing conc. HCl (35 ml), NH_4Cl (2 g), $K_2S_2O_7$ (1·5 g) and H_2SeO_3 (1·5 g). The ppt. is washed with 2% (w/v) NH_4NO_3 soln. instead of *N* HCl. Under these conditions, 0·2 to 30 mg of Nb_2O_5 or Ta_2O_5, or both, can be quant. separated from mixtures containing 100 mg each of the oxides of Sc, Y, Ce, V, Mo, Fe, Al, Sn, Pb and Bi with one pptn., and from 30 mg of TiO_2 and 50 mg each of Sb_2O_3, Sb_2O_5 or ThO_2 with three pptn. One pptn. ensures separation from 50 mg of U^{6+} when present alone, otherwise three pptn. are required. Tungsten and Zr interfere, although the latter can be tolerated when the contents of Ta and Nb are small. The reproducibility and accuracy for tantaloniobate ores are high.
W. J. BAKER

952. **Determination of oxygen in titanium-manganese alloys.** D. F. Wood and J. A. Oliver (Res. Dept., I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Analyst*, 1959, **84**, 436–439.—Since the addition of tin to the platinum bath in the semi-micro vacuum fusion procedure has removed difficulties caused by the "gettering" effect of metals having high vapour pressure at high temp., the effect of its addition to overcome similar difficulties caused by the Mn in Ti - Mn alloys was investigated. A substantial increase in the recovery of O was observed, but results were erratic. It was found that absorption of the CO formed by reaction of the evolved O with the graphite crucible occurred during the volatilisation of the Mn, and was not the result of absorption by Mn deposited on the furnace wall. If the sample (30 mg) with 10 mg of tin is placed in the crucible at 1500° and the Mn is allowed to volatilise (10 min.) before the temp. is raised to 1800° to effect combination of O and C, the results are satisfactory and within the limitations of the vacuum fusion method, *viz*, ±0·01% at the 0·1% level. The procedure should be applicable to other metals alloyed with Mn, *e.g.*, Zr, Nb and Ta. A. O. JONES

953. **Tentative methods for the determination of the sulphur content of carbon black.** American Society for Testing Materials. *A.S.T.M. Standards*, 1958, Part 9, 1855–1856.—*Method A*—The dried sample (0·5 g) is weighed into the fuel capsule crucible of a Parr oxygen bomb calorimeter. The crucible is placed in the loop electrode and a fine wire attached. Water (5 ml) is placed in the bomb cylinder, which is then assembled and filled with O to a pressure of 30 atm. The calorimeter bucket, containing 2 litres of water, is placed in the calorimeter and after stirring for 2 min. the water temp. is recorded, the ignition circuit closed and the rise in temp. observed (3° to 5° F). After 10 min. the pressure is released, the contents of the bomb are filtered, and conc. HCl (5 ml), satd. picric acid soln. (5 ml) and 10% $BaCl_2$ soln. (10 ml) are added. The ppt. is filtered off, ignited at 925° C and weighed as $BaSO_4$. *Method B*—The dried sample (1·0 g) is weighed into a combustion boat. Two 400-ml flasks are one-third filled with HCl - water (2:13). Starch indicator and sufficient KIO_3 - KI soln. (**I**) (1·1125 g of KIO_3, 100 g of KI and 5 g of KOH per litre) are added to give a perceptible colour. A coarse fritted-glass gas-dispersion tube is placed in one flask (the other is used as a colour reference standard) and connected to a combustion tube maintained at 1425° C. The boat is placed in the hot part of the tube and the tube is swept with O at the rate of 1 litre per min. The gases evolved pass into the HCl soln., which is then titrated with **I** to a permanent blue colour.

REV. CURR. LIT. PAINT COL. VARN.

954. **Determination of sulphur in yellow phosphorus.** E. E. Zussor. *Soobshch. o Nauch.-Issled. Rabot. i Novoĭ Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 80–82; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,107.—Place 10 ml of water in a dish and weigh; dry 1 to 2 g of the yellow P with filter-paper, ethanol and diethyl ether, add it to the water and re-weigh. Then transfer the sample to a beaker, add 50 to 60 ml of a satd. soln. of Br in HNO_3 (sp. gr. 1·40), heat at 70° to 80°, and evaporate to a small vol.; add Na_2CO_3 in portions (3·6 g per g of P) and evaporate to dryness; add 10 to 15 ml of conc. HCl and again evaporate to dryness; repeat this step twice. To the residue add 2 to 3 ml of HCl and 50 to 60 ml of water, boil for 5 min. and filter. Dilute the filtrate to 250 ml, add 7 ml of conc. HCl, heat to boiling, add 50 ml of boiling 2% $BaCl_2$ soln. dropwise, and heat for 4 hr. without boiling. After 12 hr. filter off the ppt. of $BaSO_4$, wash it with hot water, ignite and weigh. The method is reasonably accurate.

C. D. KOPKIN

955. **Application of ion-exchange resin to the Lunge method for the determination of sulphur in ores.** M. Ionescu, A. Demetrescu and E. Mitran. *Rev. Minelor, Bucharest*, 1958, **9**, 473–475.—A cation-exchange resin (a polymeric phenolsulphonate) is used to remove heavy-metal ions in the Lunge method for the determination of S as SO_4^{2-}. The lengthy double pptn. of heavy metals with aq. NH_3 is thus avoided. The ore is decomposed by mixing it with an oxidant and Br. The residue is treated with 0·3 *N* HCl (50 ml) and, if it is not completely dissolved, H_2O_2 (5 to 6 drops) is added to the hot soln. The soln. is filtered hot and the filter is washed 5 or 6 times with hot water. The filtrate is passed through the exchange column at the rate of 80 to 120 drops per min. (according to the heavy-metal-ion content of the sample). When the liquid reaches the resin level 25 ml of 0·1 *N* HCl is added in a thin stream to avoid mixing the soln. The addition is repeated (50 ml) and a final washing with water (50 ml) ensures complete passage of SO_4^{2-} into the receiving vessel. The SO_4^{2-} are pptd. with $BaCl_2$ soln. and the determination is continued according to the Lunge method. An accuracy of within 0·2% is reported. CHEM. ABSTR.

956. **Ultra-violet spectrophotometric determination of hydrogen sulphide and carbon disulphide in gases.** E. Geiger, H. Nobs and P. Halasz (Steckborn Kunstseide A.-G.). *Helv. Chim. Acta*, 1959, **42** (4), 1345–1350.—Hydrogen sulphide and CS_2 in gases (*e.g.*, air) are absorbed in methanolic KOH soln.; CS_2 yields K methylxanthate with an extinction max. at 302 mμ and H_2S yields K_2S, with an extinction max. at 229 mμ. Extinction coefficients were found to depend on the solvent, the mean values in methanolic KOH being 7560 at 229 mμ and 17,090 at 302 mμ for CS_2, and 8100 at 229 mμ for H_2S. *Procedure*—A measured volume of gas (≃4 litres) is drawn through 3 ml of *N* methanolic KOH and then through 2 ml of water, and the two absorbents are mixed and made up to 100 ml with water. The extinctions are measured at 229 mμ and 302 mμ against a reagent blank. If m_1 and m_2 are the extinctions at 302 mμ and 229 mμ, respectively, the concn. (mg per litre in the soln.) of CS_2 is given by $4{\cdot}455\,m_1$ and that of H_2S by $(4{\cdot}207m_2 - 1{\cdot}861m_1)$. J. L. PROSSER

957. **Determination of free and combined hydrogen sulphide in aqueous solutions of alkaline sulphides.** J.-A. Berger and J. Dauphin (Lab. de Chim. Pharm., Fac. de Méd. et de Pharm., Clermond-Ferrand, France). *Bull. Soc. Chim. France*, 1959, (4), 618–619.—The possibility of determining free and combined H_2S by bubbling the sample soln. with an inert gas is critically examined. Combined H_2S at pH 6·5 to 10 is virtually all HS^-. It is shown theoretically that a continuous evolution of H_2S is to be expected together with a continuous change of pH, and an expression is derived for the change of pH with time. Experimental work confirms this expression, though the rate of evolution is slightly lower than the theoretical owing to imperfect equilibration of the gas bubbles. It is concluded

that the bubbling technique of Dienert is adequate if the 15-min. bubbling is fast enough, and free H_2S is considered to be eliminated when the pH reaches 9·3 (achieved in 30 min. with the low rate of flow used in these experiments). The technique of extraction of H_2S under vacuum gives a high result. With mixtures of H_2S and NaHS, a determination of pH and total S is sufficient. With mineral waters the method may be affected by buffers present in the soln. E. J. H. BIRCH

958. **Determination of sulphuric acid in copper plating baths.** M. R. Verma, V. M. Bhuchar, S. K. Mathur and S. S. Sharma (National Physical Lab., Hillside Road, New Delhi, India). *Chemist Analyst*, 1959, **48** (2), 41.—The interference of Cu^{2+} in the titration of H_2SO_4 is prevented by the formation of a soluble copper oxalate complex which does not hydrolyse, even on the addition of alkali, provided that oxalate is present in a molar ratio of at least 3 to 1 with respect to copper. An aliquot of the plating solution containing about 2 milli-equiv. of acid is treated with 5 ml of 1 *M* K oxalate (neutralised to phenol red). The appropriate amount of 0·1% indicator soln. is added and the acid titrated directly with 0·1 *N* NaOH. The indicator may be either the sodium salt of phenol red (2 drops), or *o*-coumaric acid in 50% aq. ethanol (10 drops), or Na 2-naphthol-3:6-disulphonate (10 drops). Ultra-violet light must be used with the last two indicators. Typical results are tabulated. S. M. MARSH

959. **Determination of sulphuric acid in an acetylating mixture.** Tetsuro Murakami (Kogakuin, Kashiwagi, Shinjuku-ku, Tokyo). *Japan Analyst*, 1959, **8** (2), 103–106.—Since $BaSO_4$ and $PbSO_4$ are insol. in acetic acid, H_2SO_4 ($\simeq 1\%$) in an acetylating mixture is titrated with 0·1 *M* Ba or Pb acetate in glacial acetic acid. By the use of crystal violet (0·5% in acetic acid) as indicator, the end-point coincides with that obtained by the potentiometric method. Water obscures the end-point, but this effect is eliminated by the addition of acetic anhydride. Acetic anhydride in the acetylating mixture can be determined by the method of Ellerington and Nicholls (*Analyst*, 1957, **82**, 233). K. SAITO

960. **Determination of sulphate ions by the benzidine method.** Eiiti Minami and Kunihiko Watanuki (Chem. Dept., Fac. of Sci., Tokyo Univ., Hongo). *Japan Analyst*, 1959, **8** (2), 129–131.—The amount of adsorbed benzidine hydrochloride on pptd. benzidine sulphate depends on the washing agent. Acetone is better than ethanol. The solubility of the sulphate is markedly increased by the presence of Cl^-. The washed ppt. is transferred to a flask with hot water and boiled with NaCl (0·5 g) before titrating with 0·1 *N* NaOH. K. SAITO

961. **Turbidimetric titration of sulphate ions. Colloid chemical consideration of the shape of the titration curve.** Masaji Miura and Tomoya Nagakane (Fac. of Sci., Hiroshima Univ., Senda-machi). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (1), 53–55.—The photometric titration curve of K_2SO_4 with $BaCl_2$ (0·0025 to 0·01 *M*) was examined in the presence of varied amounts of ethanol, KCl and $FeCl_3$. The plot of extinction *vs.* the molar ratio of Ba^{2+} to SO_4^{2-} is convex upwards in $<25\%$ (v/v) ethanol, then becomes linear with increase in concn. of ethanol, and then becomes concave in $>35\%$ ethanol. This appears to be due to the decrease in particle size of the ppt., due to the decrease in solubility of $BaSO_4$ with increase in concn. of ethanol. Near the end-point the ppt. coagulates, causing an increase in transmission. This explanation was confirmed by the addition of KCl or $FeCl_3$ to the K_2SO_4 soln., which increases the solubility of $BaSO_4$. K. SAITO

962. **Determination of sulphate sulphur in superphosphate.** L. V. Vladimirov and M. N. Shul'gina. *Soobshch. o Nauch.-Issled. Rabot. i Novoĭ Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 15–20; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,108.—To 2·5 g of superphosphate add 15 ml of a satd. soln. of Na_2CO_3, 75 ml of water and 10 ml of 10% $CaCl_2$ soln.; boil for 1 hr., periodically adding water to maintain the vol., cool, and make up to 250 ml. Filter, neutralise 25 ml of the filtrate with dil. HCl (1:1) to methyl red, add a further 1 or 2 ml of HCl, and boil for 10 min.; cool, neutralise with 10% aq. NH_3 to litmus, make up to 250 ml and filter. To 100 ml of filtrate add 20 ml of a mixture (1:1) of 10% KI soln. and HCl previously decolorised with 1 or 2 drops of 0·05 *N* $Na_2S_2O_3$; set aside for 5 min. in the dark, dilute to $\simeq 150$ ml and titrate with 0·05 *N* $Na_2S_2O_3$, with starch as indicator. This volumetric method is 2 to 2·5 times as rapid as the gravimetric method; the relative error is $\simeq 0{\cdot}5\%$. C. D. KOPKIN

963. **Rapid method for the determination of selenium in pyrites, residues, elementary sulphur and slimes from sulphate production.** F. N. Kel'man. *Soobshch. o Nauch.-Issled. Rabot. i Novoĭ Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 68–75; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,111.—Place the sample in a boat in a quartz tube in a tube furnace, and ignite at 750° in a current of air containing oxides of nitrogen. The Se is converted into SeO_2, which passes through a condenser and is trapped on cotton-wool plugs in a crimped tube. After the ignition, transfer the cotton wool to a beaker, and wash out the crimped tube and condenser into the same beaker (total vol. <180 ml). Neutralise the soln. to phenolphthalein with aq. NH_3, add 2 or 3 drops of HNO_3 (sp. gr. 1·40) to decolorise the soln., add a further 3 ml of HNO_3 and 3 to 7 g of urea and heat to 80°; after 10 min. cool, add KI, set aside in the dark for 3 to 5 min., then add 120 ml of water and titrate the iodine (liberated in amount equivalent to the Se present) with $Na_2S_2O_3$ soln. in the presence of starch; TeO_2 and As_2O_3 do not interfere. The determination takes 1·5 to 2 hr.; the results agree well with those by other methods. C. D. KOPKIN

964. **Separation of tellurium from iodide with anion-exchange resin.** Mariko Inarida (Chem. Dept., Fac. of Sci., Tokyo Univ., Hongo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (4), 399–400.—Since TeO_3^{2-} are quant. adsorbed from soln. in HCl (3 to 12 *N*) by a column (1 cm × 5 cm) of Amberlite IRA-400 (Cl) (80 to 100 mesh) and TeO_4^{2-} pass through (*cf.* Kimura *et al.*, *Anal. Abstr.*, 1959, **6**, 140), a satisfactory separation of iodide from Te can be achieved. The sample soln. containing Te and iodide in 4 *N* HCl is passed through the column, which is then washed with 4 *N* HCl. All the TeO_4^{2-} are found in the eluate. The column is then eluted successively with 0·1 to 1 *N* HCl and 10 *N* HCl, to collect TeO_3^{2-} and I^-, respectively. K. SAITO

965. **Colorimetric method for the estimation of tervalent chromium.** M. R. Verma, V. M. Bhuchar, K. C. Agrawal and R. K. Sharma (Nat. Phys. Lab., Hillside Road, New Delhi, India). *Mikrochim. Acta*, 1959, (5), 766–769 (in English).—The method described depends on the use of Chenta acid (1:2-diaminocyclohexane-NNN'N'-tetra-acetic acid) (**I**) in complexometric determinations (Přibil *et al.*, *Coll. Czech. Chem. Commun.*, 1953, **18**, 43). Preliminary work indicated that a soln. of the disodium salt of **I** on being boiled with Cr^{III} gives a reddish-violet soln., which is stable over a pH range from 2 to 6, and has an absorption max. at 540 to 542 mμ. Under these conditions dichromate soln. do not undergo reduction. The complex obeys Beer's law in the concn. range 4 to 80 μg per ml, and the colour is stable for one week. D. F. PHILLIPS

966. **Colorimetric determination of molybdenum as the thiocyanate complex.** B. I. Nabivanets. *Ukr. Khim. Zhur.*, 1958, (6), 635–645; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,241.—Thiourea, ascorbic acid, $SnCl_2$ and KI were tested as reducing agents and the effect of an excess of reducing agent on the colour of the thiocyanate complex of Mo was studied. It was shown that the colour of the complex was more stable in H_2SO_4 than in HCl. The best reducing agent for max. colour development and stability was ascorbic acid. A determination of Mo in WO_3 with thiourea as the reducing agent is described; ascorbic acid is used as the reducing agent for the determination of Mo in steel. K. R. C.

967. **Turbidimetric micro-determination of molybdenum with ethylenediamine dihydrochloride.** G. S. Deshmukh, S. V. Tatwawadi and M. S. Vaikuntam (Chem. Dept., Hindu Univ., Benares, India). *Mikrochim. Acta*, 1959, (5), 791–792 (in English).—Aliquots of an aq. soln. of ethylenediamine dihydrochloride (**I**) (0·5 to 0·005 *M*) were mixed with known amounts (0·1 to 3 ml of 0·0002 to 0·005 *M*) of ammonium molybdate soln. and diluted to 5 ml with ethanol. Full development of a fine white turbidity required about 10 min. The extinction was measured with a Spekker absorptiometer, with a violet filter having max. transmission at 430 mμ. The extinction was found to be directly proportional to the concn. of Mo^{VI} within the range of 5 to 100 p.p.m. By using this calibration curve ≃5 p.p.m. of Mo in an unknown sample was determined when an excess of **I** was added to the sample in the presence of 80% of ethanol. Only cations that either formed complexes with Mo or were pptd. as basic salts interfered. D. F. PHILLIPS

968. **New volumetric method for the determination of molybdenum (VI). Use of hydrazine sulphate as reductant.** G. Gopala Rao and M. Suryanarayana (Dept. of Chem., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1959, **168** (3), 177–181 (in English).—Sexavalent Mo is reduced with hydrazine sulphate in 1 to 2 *M* HCl at 100° for 10 min. and then determined volumetrically as Mo^{V} with standard $Ce(SO_4)_2$ soln. in 4 *N* HCl (in the presence of 5 ml of syrupy H_3PO_4 for 50 ml of soln. and of diphenylbenzidine as indicator), or in 3 *N* HCl (in the presence of 5 ml of syrupy H_3PO_4 for 50 ml of soln. and of ferroin as indicator), or with standard sodium vanadate soln. in 8 *N* H_2SO_4 (N-phenylanthranilic acid as indicator) or in 4 *N* acid (diphenylbenzidine as indicator) in the presence of 5 ml of syrupy H_3PO_4 and 1 ml of 0·1 *M* oxalic acid as catalyst. The accuracy with 0·1 to 0·4 millimole of Mo^{VI} is within ±0·2% relative. J. P. STERN

969. **Rapid method of determining molybdenum in molybdenum concentrates.** A. V. Vinogradov and T. I. Evseeva. *Zavod. Lab.*, 1959, **25** (5), 550–552.—The method is based on the possibility of precipitating Mo with 8-hydroxyquinoline in 0·1 *N* H_2SO_4. The sample (0·1 g) of concentrate is fused with 6 g of $K_2S_2O_7$ at 650° to 700°, and the melt is boiled with water containing 5 ml of dil. H_2SO_4 (1:1). The soln. is filtered, the residue being washed with hot water containing 2 ml of conc. H_2SO_4 in 100 ml, and evaporated to a vol. of 50 ml. It is then treated with 10 ml of 0·05 *M* EDTA (to form a complex with Fe), aq. NH_3 (to neutralise, with methyl red as indicator), 7 ml of *N* H_2SO_4, and water (to give a vol. of 100 ml). The boiling soln. is treated with 10 ml of a 3% soln. of 8-hydroxyquinoline in H_2SO_4, neutralised with aq. NH_3 to a pH of between 4 and 5, and the ppt. is filtered off in a sintered glass crucible after 1 to 2 min. and washed with hot water. The ppt. is dried at 120° to 140° and weighed. If W is present, 5 to 10 ml of 10% oxalic acid soln. is added after the pptn., and the soln. is boiled for a further 2 min. before the filtration.

G. S. SMITH

970. **Determination of molybdenum in fused mixtures of sodium, zirconium and uranium fluorides.** U.K.A.E.A. (Res. Group, Woolwich Outstation, Royal Arsenal, London, S.E.18). AERE-AM 26, 1959, 3 pp.—The method is based on standard analytical practice and is written with full details by A. Parker.

971. **Determination of tungsten in Zircaloy-2.** U.K.A.E.A. (Res. Group, Woolwich Outstation, Royal Arsenal, London, S.E.18). AERE-AM 13, 1959, 3 pp.—The method is based on that of Greenberg (*Anal. Abstr.*, 1957, **4**, 3934) and is written with full details by E. Booth.

972. **Determination of tungsten as tristri-*n*-butylammonium 12-tungstophosphate.** C. C. Miller and D. H. Thow (Chem. Dept., Univ., Edinburgh). *Analyst*, 1959, **84**, 440–446.—The soln. (≃50 ml) containing 10 to 200 mg of W as $NaWO_4$ and <85 mg of PO_4^{3-} as Na_2HPO_4 is adjusted to pH 2 with 6 *N* HCl, 4 ml is added in excess and the mixture is diluted to 100 ml. At near b.p. 10 ml of 2% w/v tri-*n*-butylammonium chloride soln. (prepared by shaking tri-*n*-butylamine with just enough dil. HCl to neutralise it) is added and the ppt. is coagulated by heating. The ppt. is collected in a sintered glass or porcelain crucible, washed with the least amount of tri-*n*-butylamine soln. (0·1% in 0·1 *N* HCl) and finally with a little cold water. The ppt. is heated at 210° for 2 hr. and finally weighed. Considerable amounts of the chlorides of Ca, Co, Cu, Mn and Ni and $Pb(NO_3)_2$ and large amounts of NaCl, $NaNO_3$ and Na_2SO_4 can be tolerated. A. O. JONES

973. **Volumetric determination of tungsten and uranium.** R. Weiner and P. Boriss (Lab. f. Elektronenmikroskopie, Univ., Innsbruck, Austria). *Z. anal. Chem.*, 1959, **168** (3), 195–198.—The solubility product at pH 6 of Ag_2WO_4 is $6{\cdot}52 \times 10^{-11}$ and Ag_2WO_4 is unsuitable for the gravimetric determination of W. Tungsten cannot be determined by the use of a silver reductor. However, U (2×10^{-3} mole per litre) can readily be determined to within ±0·13% by reduction in a silver reductor in 4 *N* HCl at 60°, followed by titration with $K_2Cr_2O_7$, with diphenylamine as indicator. The silver reductor appears to offer no advantages over the lead reductor.

J. P. STERN

974. **Rapid colorimetric method for the determination of uranium.** P. Blanquet. *Chim. Anal.*, 1959, **41** (6), 247–252.—The dibenzoylmethane method of Yoe *et al.* (*cf. Anal. Abstr.*, 1954, **1**, 80) has been studied and optimum conditions established. The procedure finally devised is applicable to minerals of unknown composition. *Procedure*—For U_3O_8 contents of $<0{\cdot}5\%$, weigh 250 mg of sample, ground to pass a 50-micron sieve, into a 50-ml beaker, add H_2SO_4 (1 ml) and $HClO_4$ (0·5 ml), and evaporate to fumes and finally to dryness. Add H_2SO_4 (0·5 ml) and $HClO_4$ (0·5 ml), repeat the evaporation, and fuse the residue with $2 \pm 0{\cdot}1$ g of $KHSO_4$. Cool and extract the melt by gentle heating with 5 ml of 5% ethanol and 5 ml of "ferric solution" (300 ml of "commercial" $FeCl_3$ soln., 45° Bé, 15 ml of 85% H_3PO_4 and 500 ml of "commercial" $HClO_4$, diluted to 2 litres). Cautiously add 15% Na_2CO_3 soln. (25 ± 1 ml) and digest at a temp. near the boiling-point for 15 min. Transfer to a 50-ml flask, cool, dilute to volume, mix, and filter through a dry paper, rejecting the first 5 or 10 ml. Pipette 5 ml into a 25-ml flask and add 1·0 ml of tartaric acid soln. (50%) and dilute to volume with the reagent soln. (dibenzoylmethane 2 g, EDTA 12·5 g, and pyridine 500 ml, diluted to 1 litre). Mix, set aside for 30 min., and measure the extinction at 415 mμ in 3-cm cells against a reagent blank prepared similarly. For U_3O_8 contents between 0·5 and 2·5%, the test soln. may be diluted with the blank soln.; for contents $>2{\cdot}5\%$, dilution stages earlier in the procedure are recommended.
T. R. Andrew

975. **Analysis of uranium solutions using anion exchange in nitrate media.** O. A. Vita, C. F. Trivisonno and C. W. Phipps (Goodyear Atomic Corp., Portsmouth, Ohio). *U.S. Atomic Energy Comm.*, Rep. GAT-283, 1959, 14 pp.—A method for the separation of milligram amounts of U from large amounts of other elements has been developed. In conjunction with the basic peroxide colorimetric determination, this is a reliable procedure for the analysis of many samples which cannot otherwise be handled conveniently. The procedure consists in adsorption of the U from a conc. aluminium or nickel nitrate soln. on to an anion-exchange resin, elimination of the impurities and the adsorption soln. by washing with satd. $NaNO_3$ soln., elution of the U with dil. HNO_3 and determination of the U by the basic peroxide colorimetric method. The results indicate that a limit of error of 1·9% per determination and a bias of $-0{\cdot}04 \pm 0{\cdot}60\%$ at the 95% confidence level can be achieved.
Nucl. Sci. Abstr.

976. **Spectrophotometric method for uranium using tributyl phosphate and dibenzoylmethane.** J. L. Long and L. F. Grill (Dow Chemical Co., Rocky Flats Plant, Denver, Colo., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. RFP-78, 1957, 20 pp.—Uranyl nitrate is extracted into tributyl phosphate, where, after buffering with tri(hydroxymethyl)-methylamine and adding ethanol, the yellow uranium dibenzoylmethane complex is formed. The extinction of the soln. is measured at 405 mμ. The molar extinction is 16,600. The method is suitable for the rapid determination of microgram amounts of U in the presence of milligram amounts of interfering ions.
Nucl. Sci. Abstr.

977. **Spectrophotometric extraction methods specific for uranium.** W. J. Maeck, G. L. Booman, M. C. Elliott and J. E. Rein (Phillips Petroleum Co., Idaho Falls, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1130–1134.—In the two procedures given, based on the method of Maeck *et al.* (*cf. Anal. Abstr.*, 1958, **5**, 3718), U, as tetrapropylammonium uranyl trinitrate, is quant. separated from high concn. of many other ions by extraction into *iso*butyl methyl ketone from a 1 to 2 *N* acid-deficient $Al(NO_3)_3$ salting soln. (prep. described). (*i*) Milligram amounts (0·5 to 12 mg) of U in the organic phase are then determined directly by measuring the extinction of $UO_2(NO_3)_3^-$ at 452 mμ in a 1-cm Corex cell containing a Teflon spacer. (*ii*) Microgram amounts of U (0·8 to 75 μg) can be determined in the organic phase by increasing the colour sensitivity by the addition of dibenzoylmethane ($\simeq 0{\cdot}02\%$, w/v) in a 5% (v/v) soln. of ethanol in pyridine, and then measuring the extinction of the complex at 415 mμ in a 5-cm Corex cell. For both (*i*) and (*ii*) the blank is prepared by substituting 1 *N* HNO_3 for the sample aliquot ($>0{\cdot}5$ ml). Interference by Th and Ce^{4+} is avoided in (*i*) by increasing the vol. of reagent or by limiting the sample aliquot, and in (*ii*) by one or two treatments with specified scrubbing soln. before adding the chromogenic agent. The coeff. of variation is $<1\%$ at the 10-mg and 25-μg levels.
W. J. Baker

978. **Potentiometric determination of small amounts of uranium(VI) in uranium dioxide.** J. Minczewski, R. Przytycka and Ł. Kohman (Inst. of Nuclear Res., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (1), 27–32.—Uranium dioxide containing traces of UO_3 is converted into UO_2F_2 and UF_4 and, after the addition of K Na tartrate is titrated potentiometrically with $Ti_2(SO_4)_3$ under CO_2 at 70°.
H. Dmowska

979. **A γ-counting method for the determination of uranium-235 in enriched uranium.** U. L. Upson and D. G. Miller (General Electric Co. Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-47867 (Rev.) (Del.), Feb., 1959. Decl. with deletions May, 1959. 24 pp.—The method has been applied to the determination of ^{235}U, at $\simeq 93\%$ enrichment level, in uranium - aluminium alloys. The counting is performed on liquid samples obtained from dissolution of the alloy without chemical separation. The accuracy of an isotopic analysis by this method is dependent on the use of accurate standards and on the accuracy of the separate analysis for total U. For samples in which the total U content is accurately known, as in the case of soln. of pure U, weighed as U_3O_8, a precision of $\pm 0{\cdot}3\%$ at the 95% confidence level can be attained. For samples in which the total U content is determined by chemical analysis, the over-all precision ranges from $\pm 0{\cdot}4\%$ to $\pm 0{\cdot}8\%$ of the total U content. While γ-counting is not competitive with the mass spectrometer in precision, its virtue lies in the relative speed and economy with which samples can be evaluated, and in the fact that a mass spectrometer is not always readily available.
Nucl. Sci. Abstr.

980. **The uranium spectrum between 3500 A and 5500 A with associated isotopic shifts.** E. W. T. Richards and M. D. Crew (U.K.A.E.A., Res. Group, Harwell, Berks.). AERE-R 2941, 1959, 202 pp.—Measurements of 7700 lines in the region 3500 to 5500 A are recorded together with their intensities and in most cases the associated isotope shift. The intensities as measured in the U.S.A. by Kiess and Harrison are also given.
N. E.

981. **Separation of uranium and bismuth with ion-exchange papers.** H. T. Peterson, jun. (999 DeWitt St., Valley Stream, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1279–1280.—Small amounts ($\simeq$40 μg) of U (as UO_2^{2+}) can be separated from $\simeq$10 mg of Bi by ascending chromatography on paper impregnated with Amberlite IRA-400 (Cl^- form) in $\simeq$45 min. with H_2O as solvent. Smaller amounts (*e.g.*, 10 μg of each element) can be separated similarly on paper impregnated with Amberlite IR-45 (OH^- form). The U spot is revealed with 3% $K_4Fe(CN)_6$ soln. and the Bi spot with a satd. basic soln. of Na_2S. Conventional separations are incomplete even after development for 10 hr.

W. J. Baker

982. **Applications of X-ray fluorescence spectrography to the determination of uranium and thorium.** R. W. Jones (Atomic Energy of Canada Ltd., Chalk River, Ont.). Atomic Energy of Canada Ltd., Rep. CRDC-843, 1959, 20 pp.—Several methods are described. In pure soln. the sensitivity for these elements is 5 to 10 p.p.m. For soln. containing large concn. of impurities, Sr is added as an internal standard. The precision and accuracy of the determinations are $\simeq$1% when working in the optimum concn. range. Nucl. Sci. Abstr.

983. **Spectrophotometric determination of magnesium in uranium.** N. N. Krot, A. P. Smirnov-Averin and A. G. Kozlov. *Zhur. Anal. Khim.*, 1959, **14** (3), 354–355.—Neutralise the test soln. (containing <5 μg of Mg) in a quartz vessel, dilute to 20 to 25 ml and add acetate buffer soln. [Na acetate soln. (380 ml) and conc. HCl (10 ml), diluted to 1 litre] (5 ml). Heat the soln. to 70° to 90° and slowly add a 10% soln. of 8-hydroxyquinoline in acetone to precipitate the uranium and interfering impurities (Fe^{3+}, Cr^{3+}, Co^{2+}, Al^{3+}, MoO_4^{2-} and Ni^{2+}). Coagulate the ppt. by boiling for 3 to 5 min., then filter off and wash it with hot H_2O. Collect the filtrate and washings, remove the excess of 8-hydroxyquinoline and the traces of metals remaining in soln. with small portions (10 to 15 ml) of $CHCl_3$. Filter the aq. phase, adjust the pH to 9·8 to 10·6 with aq. NH_3, add a 0·1% methanolic soln. of Eriochrome black T (1 ml), dilute to 50 ml and measure the extinction at 250 mμ. Compare with a standard curve. Determinations can be carried out in HCl, HNO_3, H_2SO_4 or acetic acid media; up to a 20-fold excess of PO_4^{3-} does not interfere. The method can be used to determine >0·005% of Mg in uranium with a coeff. of variation of $\pm$3%. K. R. C.

984. **Determination of impurities in uranium by X-ray fluorescence spectrometry.** R. W. Jones (Atomic Energy of Canada Ltd., Chalk River, Ont.). Atomic Energy of Canada Ltd., Rep. AECL-822, 1959, 12 pp.—The method described is comparable in sensitivity and accuracy to existing spectrophotometric procedures for Cr, Fe, Ni and Zr (the impurity elements examined), but is superior because of its speed. The procedure should be applicable to the determination of numerous other impurity elements in uranium. Nucl. Sci. Abstr.

985. **Determination of neptunium by controlled-potential coulometry.** R. W. Stromatt (General Electric Co., Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-59447, 1959, 19 pp.—The method is accurate and precise. The electrolysis cell contained a platinum working electrode at which the Np^V - Np^{VI} couple was titrated; 1·0 *N* H_2SO_4 was used as the titration medium. The coeff. of variation ranged from 1% for 20 μg to 0·05% for <1 mg of Np in the cell. By using the utmost care, as little as 2 μg of Np can be detected and 7 μg can be titrated with a coeff. of variation of 6%. Under ordinary conditions, large excesses of Pu and Fe (as well as U and other elements) can be tolerated or their interference calculated. The Np is first oxidised to Np^{VI} with Ce^{IV}, the Np^{VI} and excess of Ce^{IV} are electrolytically reduced to Np^V and Ce^{III}, followed by the coulometric oxidation of the Np^V to Np^{VI}. The integrated current in the last step is used to calculate the concn. of the Np. A soln. can be analysed for the concn. of the Np oxidation states with the coulometer, with the platinum electrode cell. This analysis depends on the irreversibility of the Np^{IV} - Np^V couple; that is, Np^{IV} is not oxidised at a significant rate during the coulometric reduction of Np^V. The Np^{VI} is determined by a coulometric reduction to Np^V. The Np^V is then oxidised coulometrically, the difference between these two titrations giving the concn. of Np^V. The total Np concn. is determined as described above and the Np^{IV} concn. is calculated from the difference between the concn. of Np^V plus Np^{VI} and the total Np concn. All substances that undergo oxidation or reduction at the applied potentials interfere and must be removed or masked in order to carry out the determination. The accuracy and precision depend on the total Np concn. and on the concn. of the various Np oxidation states present. Although the precision and accuracy are not as good as for the determination of the total Np, this method is useful in the study of Np chemistry.

Nucl. Sci. Abstr.

986. **Precise determination of plutonium by potentiometric titration.** G. R. Waterbury and C. F. Metz (Univ. of California, Los Alamos Sci. Lab., N. Mex., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1144–1148.—From 200 to 400 mg of Pu in a weighed aliquot ($\simeq$2 to 4 g) of sample soln. is quant. oxidised with fuming $HClO_4$, and Pu^{6+} are then reduced to Pu^{4+} with a slight excess of 0·5 to 0·7 *M* $(NH_4)_2SO_4.FeSO_4$ soln., the excess of which is back-titrated with Ce^{4+}. A modified Sargent - Malmstadt differential potentiometric automatic titrator having a small-volume burette is used (*cf.* Waterbury, *Ibid.*, 1959, **31**, 1138), but a manual titration can also be made. Weight burettes are used for measuring sample aliquots and for adding the titrant. The method is applicable to soln. of plutonium metal or alloy (even containing Fe) in HCl, HNO_3 or $HClO_4$, but not to soln. containing PO_4^{3-} or SO_4^{2-}. The error is almost negligible for high-purity metal; mean values of 99·98%, with a standard deviation of 0·02% (11 determinations), were obtained for a content of Pu of 99·98%, calculated after deducting the total trace impurities. There is, however, serious interference by Cr, Au, Mn, V and Pt, and an initial quant. separation of Pu would then be necessary. The detection of the end-point, the effect of foreign ions, and the separation of Pu are discussed. W. J. Baker

987. **Determination of plutonium in irradiated uranium fuel solutions by controlled-potential coulometry.** R. M. Peekema and F. A. Scott (General Electric Co., Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-58491, 1958, 29 pp.—The method was applied to the determination of Pu in soln. containing from 0·05 to 50 g of Pu per litre. Electronic equipment, titration cells and procedures are

described. The procedures are simple and the equipment dependable enough for routine control laboratory application. The method has been applied to soln. containing ≃20 g of Pu per litre with a coeff. of variation of ±0·05% and with an accuracy within 0·1%. The method has also been applied to the direct determination of the Pu content of soln. of irradiated natural uranium (dissolver soln.) with a coeff. of variation of ±0·5% and an accuracy of within 1·4%. The titration curves follow the Nernst equation exactly for ions that undergo reversible electrode reactions. Hence, if the concn. and $E_{\frac{1}{4}}$ of an interfering material are known, a theoretical estimation of the magnitude of the interference can readily be made. The method is remarkably free from interference. Moderate variations in the acid and in the salt content of the sample can be tolerated. There are few reversibly titratable ions that have standard electrode potentials near enough (±200 mV) to that of the Pu^{IV}-Pu^{III} couple to cause them to interfere seriously. The redox potentials of these substances, *e.g.*, Fe and Hg, can usually be shifted far enough from that of Pu to eliminate interference by performing the titration in the proper complexing medium. Several such media have been investigated and are compared. NUCL. SCI. ABSTR.

988. **The optical spectra of some rare earth and transuranic elements in the 1 to 3-micron region. II. Wavelength measurement and the spectrum of plutonium-239.** L. Bovey, E. B. M. Steers and N. Atherton (Chem. Div. A.E.R.E., Harwell, Berks.). AERE-R 2977, 1959, 12 pp.—Methods for the measurement and comparison of the wavelengths of the plutonium lines in the 1 to 3-μ region (10,000 to 3,300 cm^{-1}) are given together with an indication of the experimental accuracy. A list of the wavelengths and intensities for plutonium lines is included.

989. **Determination of fluorine by a fluorescence titration method.** M. M. Vinnik and M. L. Chepeleveteskii. *Soobshch. o Nauch.-Issled. Rabot. i Novoi Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 44–57; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,115.—For the determination of small amounts of F, make the test soln. alkaline with KOH and then add 0·01 *N* HCl till the soln. is neutral to phenolphthalein and 2 drops in excess; add ethanol (12 ml for each 5 ml of original soln.), NaCl (to saturation) and 0·35 ml of 0·1% morin soln., and titrate fluorimetrically with 0·001 to 0·002 *N* $KAl(SO_4)_2$. The end-point is determined on the assumption that the concn. of the morin complex of Al at the end-point (*X* equiv.) is independent of the original concn. of F^-; determine the value of *X* by titrating a soln. of known concn. It was assumed that the F^- were combined as Na_3AlF_6; the influence of possible deviations of the composition of the ppt. from the theoretical was eliminated by standardising the soln. against fluoride. The maximum error of the titration is 0·005 mg for 0·047 to 1·145 mg of F. When using ≃0·01 *N* soln. the end-point may be determined by the intersection of the rectilinear portions of the titration curve. A visual fluorescence titration is possible when using soln. of Al^{3+} of concn. ≮0·01 *N*: neutralise the boiling soln. to phenolphthalein with KOH soln., add 0·01 *N* HCl to decolorise the soln. and 2 drops in excess, and add sufficient ethanol to make its final concn. at the end of the titration ≃50%; add 3 to 5 g of NaCl and 30 to 35 drops of a 0·1% ethanolic soln. of morin and titrate with 0·01 to 0·05 *N* $KAl(SO_4)_2$ to a green - yellow fluorescence. A simple fluoriscope is described. The methods described are also suitable for the determination of F in distillates. C. D. KOPKIN

990. **New colorimetric determination of fluoride ions with the zirconium complex of 4-dimethylaminoazobenzene-4-arsonic acid.** Masaaki Kamada and Tomio Onishi (Fac. of Lit. and Sci., Univ., Kamoike-cho, Kagoshima). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (3), 275–280.—An aq. suspension of the Zr complex of 4-dimethylaminoazobenzene-4-arsonic acid (**I**) reacts with F^- in an HCl soln. to release an equiv. amount of **I** into solution. The excess of the complex is filtered off, and the amount of F^- is determined from the extinction of the filtrate at 500 mμ. The molar extinction coeff. of **I** increases with increase in concn. of HCl. The resulting **I** soln. is very stable and the working curve is linear for <10 p.p.m. of F^-. There is no interference from S^{2-}, $S_2O_3^{2-}$ (<2 p.p.m. each), Al, SO_3^{2-}, NO_2^- (<10 p.p.m. each), Ca, SO_4^{2-} or SiO_3^{2-} (<100 p.p.m. each). This method is suitable for the analysis of natural waters. The sample (10 ml) is shaken with the reagent suspension (2 ml for 0·2 to 10 p.p.m. of F^-; 1 ml and 2 ml of 6 *N* HCl for 0·02 to 1 p.p.m.), filtered after 15 min. and the extinction measured at 500 mμ. *Reagent*—Mix **I** soln. (0·2 g in 100 ml of 6 *N* HCl at 60° to 70°) with $ZrOCl_2$ soln. (0·1 g of $ZrOCl_2.8H_2O$ in 100 ml of 6 *N* HCl); filter after 30 min., separate the ppt. by centrifuging and wash it with *N* HCl (4 × 30 ml). Suspend the product in 500 ml of *N* HCl. The suspension is stable for several weeks. K. SAITO

991. **Separate determination of small amounts of hydrofluoric, fluorosilicic and orthophosphoric acids when present together.** M. M. Vinnik. *Soobshch. o Nauch.-Issled. Rabot. i Novoi Tekhn. Nauch. Inst. po Udobr. i Insektofungisidam*, 1958, (10), 26–36; *Ref. Zhur., Khim.*, 1959, (12), Abstr. No. 42,119.—A method is evolved for the separate determination of HF, H_2SiF_6 and H_3PO_4 when present together in condensates obtained during the production of fluorine-free phosphates. *Procedure for P_2O_5*—To an aliquot of the test soln. add 0·5 g of H_3BO_3, dilute to 50 ml, add 5 ml of reducing soln. (2 g of metol, 10 g of $Na_2SO_3.7H_2O$ and 600 ml of aq. $NaHSO_3$, containing 20 to 22% of SO_2 per litre) and 10 ml of a 5% soln. of ammonium molybdate in H_2SO_4 (1:1); set aside for 10 min., add 20 ml of 34% Na acetate soln., and measure the extinction. *Procedure for total F*—Heat an aliquot of the test soln. in a Plexiglas flask to between 50° and 70° and titrate to phenolphthalein with 0·1 or 0·01 *N* NaOH; correct for the amount of NaOH used in titrating the H_3PO_4 present. *Procedure for H_2SiF_6*—Add an aliquot of the test soln. to a Plexiglas flask containing 1·5 g of H_3BO_3, and add 35 ml of 0·15 *N* H_2SO_4 and 5 ml of 5% ammonium molybdate soln.; set aside for 5 min., add 15 ml of 8 *N* H_2SO_4 and 20 ml of $(NH_4)_2SO_4.FeSO_4.6H_2O$ soln. (100 g of salt and 450 ml of conc. H_2SO_4 in 2 litres), make up to 100 ml and measure the extinction after 15 min. C. D. KOPKIN

992. **Iodimetric assays. VII. The determination of iodine in the presence of arsenate.** L. Szekeres and M. Rady. *Magyar Kém. Lapja*, 1959, **14** (6), 249–250.—No reduction of AsO_4^{3-} by I^- could be observed when a 0·5 to 0·8 *N* H_2SO_4 soln. was employed. The I^- are oxidised to IO_3^- with BrO^- or ClO^- and the excess of reagent is subsequently

reduced by H_2O_2, ethanol, sodium formate, etc. The Winkler method can be then used, if H_2SO_4 is added. *Procedure*—The sample (0·05 to 0·005 g of I^- and $\geq$0·05 g of AsO_4^{3-}) is dissolved in water (15 ml) and 0·1 *N* KBr - Br soln. (40 ml) and $NaHCO_3$ (2·5 g) are added. After a few min., the soln. is treated with ethanol (15 ml) or 3% H_2O_2 soln. (15 ml) and heated on a water bath for 15 min. After cooling, 10 *N* H_2SO_4 (5 ml) and 2% KI soln. (30 ml) are added and the iodine is titrated with 0·05 to 0·1 *N* $Na_2S_2O_3$. G. SZABO

993. **Carrier-free separation of iodine-131 from irradiated unit I-131.** Mariko Inarida (Chem. Dept., Fac. of Sci., Tokyo Univ., Hongo). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (4), 400–401.—Irradiated unit I-131 (target material Te) is fused with KOH and dissolved in sufficient dil. HCl to produce a soln. *N* with respect to HCl and containing 1 mg of Te per ml. The product is passed through a column (1 cm × 5 cm) of Amberlite IRA-400 (Cl) (80 to 120 mesh) at a rate of flow of 1·5 ml per min. Only iodine is adsorbed on the resin in the carrier-free state, and is eluted with 6 *N* HCl (50 ml). The radiochemical yield for I is $\simeq$50% in the carrier-free state. The Te is separated by pptn. with SO_2 in 3 *N* HCl. K. SAITO

994. **Potentiometric determination of iodine and bromine.** I. Streipa. *Latv. Iauksaimniecības Akad. Raksti*, 1958, (7), 25–32; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,583.—A method is developed for the titration of Br^- and I^- with $AgNO_3$ soln. and silver and silver amalgam electrodes. The error of the determination for >2·5 μg of Br^- and >0·4 μg of I^- is 4 to 7 and 2 to 7%, respectively. By using a micro-burette calibrated to 0·001 ml, as little as 0·5 μg of Br^- and 0·1 μg of I^- may be determined; 30 to 40% of acetone and 1 or 2 drops of 0·2 *N* H_2SO_4 should be added to the soln. When Br^- and I^- are present together, as little as 1 μg of I^- per ml may be determined if the excess of Br^- is <3- or 5-fold. The end-point of the titration is not sharp. Chloride interferes in the determination of Br^-, but not in that of I^- when the ratio of Cl^- to I^- is 1:1. The procedure may be improved by ensuring the rapid coagulation of the AgBr and AgI. C. D. KOPKIN

995. **Spectrophotometric determination of iodides and bromides in oil-field brines.** A. G. Collins and J. W. Watkins (Bur. of Mines, U.S. Dept. of the Interior, Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1182–1184.—The method given is specially applicable to samples in which the ratio of Cl^- to other halides is >10. The iodides are oxidised to iodine with 10% KNO_3 soln. at pH 5 (H_2SO_4), the iodine is extracted with CCl_4, and the extinction of the organic phase is measured at 517 mμ (0·5-in. cell). The bromides in the iodine-free soln. are then oxidised to Br with HOCl, the Br is extracted with CCl_4 and the extinction is measured at 417 mμ. The sensitivity, precision and accuracy are (in mg per litre) 0·2, 0·5 and 1·4 (for I^-) and 5, 4·6 and 7·1 (for Br^-). W. J. BAKER

996. **Spectrographic determination of calcium, silicon, barium, potassium and rubidium in high-purity sodium iodide.** Yu. I. Pecherskaya and G. A. Pevtsov. *Trudy Vses. Nauch.-Issled. Inst. Khim. Reaktivov*, 1958, (22), 56–58; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,601.—A semi-quant. method is described, in which an a.c. arc is used, for the rapid control of the purity of NaI. A few drops of the test soln. are placed in a cavity in the carbon electrode; visual photometry of the lines is used. The analytical conditions are different for each element to be determined. C. D. KOPKIN

997. **Chelometric titration of manganese in ferromanganese.** R. Přibil and M. Kopanica (Chem. Inst., Czech. Acad. of Sci., Prague). *Chemist Analyst*, 1959, **48** (2), 35–36.—Manganese in ferrous alloys containing a high proportion of Mn (>40%) may be titrated directly with EDTA soln., with Thymolphthalexon [3:3′-bis-NN-di(carboxymethyl)aminomethylthymolphthalein] (**I**) as indicator. The alloy (0·25 g) is dissolved in conc. HNO_3 and conc. HCl and the soln. is diluted to 250 ml. To a 25- or 50-ml aliquot of this soln. is added 5 ml of 10% aq. hydroxyammonium chloride soln., 10 ml of 20% aq. triethanolamine soln., 30 to 35 ml of conc. aq. NH_3, and water to 150 ml. Sufficient indicator (1 part of **I** ground with 100 parts of KNO_3) is added to produce a blue colour, and the titration is carried out with 0·05 *M* EDTA (disodium salt) until the indicator becomes permanently colourless or slightly pink. Typical results are quoted. Trace amounts of Cu and Zn do not interfere and larger amounts can be masked by the addition of 50 mg of KCN. Copper may be determined photometrically on the sample soln. with Na diethyldithiocarbamate. If the sample soln. contains >25 mg of Fe per 100 ml the intense colour obscures the colour change of the indicator. S. M. MARSH

998. **Determination of rhenium in molybdenite by a colorimetric method [based on the catalytic action of perrhenates on the reaction between sodium tellurate and stannous chloride].** N. S. Poluéktov and L. I. Kononenko (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (5), 548–550.—For removal of the bulk of the Mo, the sample is sintered with a mixture of CaO and $Ca(NO_3)_2$ and the Re is extracted with water. Traces of Mo are removed from the soln. by addition of 8-hydroxyquinoline and extraction of the complex formed by Mo with $CHCl_3$. The reagent soln. is prepared by mixing 5 ml of 0·5% Na_2TeO_4 soln., 2 ml of 45% tartaric acid soln., 1·5 ml of 0·5% gelatin soln. and 1·5 ml of a soln. of 30 g of $SnCl_2.2H_2O$ in 15 ml of conc. HCl. The presence of Re causes the formation of elementary Te, the amount of which is determined colorimetrically. G. S. SMITH

999. **New sensitive method for the detection of tervalent iron.** V. Armeanu, D. Camboli and C. Iancu (Polytech. Inst., Bucharest). *Bul. Inst. Polit. Bucureşti*, 1958, **20** (2), 85–87 (in French).—The method is based on the intense red coloration produced by Fe^{3+} and β-furil monoxime (**I**). *Procedure*—Filter-paper is spotted with a drop of the test soln., dried over a bunsen flame, and a drop of a 1% ethanolic soln. of **I** is added, followed by exposure to NH_3 vapour; Bi^{3+}, Sn^{2+}, Sn^{4+}, Sb^{3+}, Sb^{5+}, AsO_3^{3-} and AsO_4^{3-} interfere in concn. >20 times, Cr^{3+} in concn. >50 times, Al^{3+} and Pb^{2+} in concn. >100 times, and Hg^{2+} in concn. >400 times that of Fe. The limit of detection is 0·32 μg of Fe, and the dilution limit is 1 in 2 × 10^5. H. SHER

1000. **Ferron as an indicator in the complexometric titration of ferric iron.** Soichiro Musha and Kin'ya Ogawa (Fac. of Engng, Univ. of Osaka Prefecture, Sakai). *Japan Analyst*, 1959, **8** (3),

161–166.—The molar ratio and the formation constant of the Fe^{III} - ferron (**I**) complex have been determined spectrophotometrically (Fe^{III}:**I** = 1:3; $K = 1 \times 10^{27}$) and its use in EDTA titrations is discussed. At pH 2·5, Fe^{III} (>50 mg) is titrated with **I** as indicator with a coeff. of variation of ≃0·5%. For smaller amounts of Fe^{III}, photometric titration at 610 mμ is suitable, the coeff. of variation being <1%. Copper and Co interfere.

K. Saito

1001. **Complexometric determination of iron.** A. A. Bashkirtseva and E. M. Yakimets (S. M. Kirov Ural Polytech. Inst.). *Zavod. Lab.*, 1959, **25** (5), 540–542.—For successful use of the volumetric EDTA method of determining Fe^{3+} in the presence of NH_4SCN or sulphosalicylic acid as indicator, the following conditions must be observed: the content of Fe should lie between 0·1 and 100 mg per 100 ml, but NH_4SCN is recommended only for the range of 0·1 to 1 mg and sulphosalicylic acid for the range of 1 to 100 mg; the indicator concn. should correspond to 5 ml of 20% NH_4SCN soln. per 100 ml or to 1 ml of 10% sulphosalicylic acid soln. per 100 ml; for determining Fe in the presence of other metals a low value of pH is necessary (1 is suitable with sulphosalicylic acid, and between 1·3 and 1·4 with NH_4SCN); the temp. of the soln. to be titrated should be between 60° and 70°. For determining Fe in various materials (magnesite, dolomite, bauxite, etc.), a suitable aliquot of the sample soln. from which SiO_2 has been removed is oxidised with HNO_3, and the boiling soln. is treated with *N* NaOH until a permanent cloudiness appears (or to the colour change of Congo red paper). Then with contents of Fe between 0·1 and 1·0 mg, 5 ml of *N* HCl is added, the vol. is brought to ≃100 ml and the temp. to between 60° and 70°, 5 ml of 20% NH_4SCN or KSCN soln. is added, and titration is carried out with 0·005 *M* EDTA (disodium salt), with the addition during the titration of small amounts of $(NH_4)_2S_2O_8$. With contents of Fe between 1 and 100 mg, the neutralised soln. is treated with 9 ml of *N* HCl and the heated soln. (≃100 ml) is mixed with 1 ml of 10% sulphosalicylic acid soln. and titrated with 0·025 or 0·05 *M* EDTA (disodium salt). No interference is caused by large amounts of Zn, Al, Mn, alkali and alkaline-earth metals, Cl^-, NO_3^- and SO_4^{2-}.

G. S. Smith

1002. **Spectrophotometric determination of iron as ferric acetate complex.** D. D. Perrin (Dept. Med. Chem., Australian Nat. Univ., Canberra). *Anal. Chem.*, 1959, **31** (7), 1181–1182.—Soln. containing 0·06 to 31 mg of Fe^{3+} can be titrated spectrophotometrically at 337·5 or 465 mμ (1-cm cell) in 2 *M* Na acetate - 2 *M* acetic acid buffer. Beer's law is valid, at either wavelength, for the ferric acetate complex over the range of 3 to 1200 p.p.m. of Fe. The error is ≃±1%. The effects of other ions are discussed; F^-, PO_4^{3-} and oxalate should be absent, or present in only low concn. The limit of detection is 2 μg of Fe in 25 ml (5-cm cell).

W. J. Baker

1003. **Selective liquid - liquid extraction of iron with 2-thenoyltrifluoroacetone in xylene. Application to homogeneous reactor solutions.** F. L. Moore, W. D. Fairman, J. G. Ganchoff and J. G. Surak (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1148–1151.—In the determination of the specific activity of ^{59}Fe in soln. of $UO_2(SO_4)_2$, the extraction of Fe^{3+} from 10 *M* HNO_3 soln. with 0·5 *M* 2-thenoyltrifluoroacetone - xylene is efficient and is more highly selective than is extraction with diethyl ether or anion-exchange from HCl soln. After washing the organic phase with 0·25 *M* HF - 0·25 *M* HNO_3 (to increase the selectivity), Fe is removed therefrom with conc. HCl. Provided that, in general, an initial pptn. of $Fe(OH)_3$ with aq. NH_3 is made and that any HF is removed or masked before the extraction step, there is excellent separation of Fe from alkali and alkaline-earth metals, rare-earth elements, Ru, Zr, Nb, I, Sn, Sb, Co, Mn, Cr, Ni, Th, Pa and U.

W. J. Baker

1004. **Rapid determination of ferric iron in hydrochloric acid with titanous chloride.** Kiyosu Nakayama (Kooriyama Plant, Hodogaya Chem. Ind. Kooriyama, Fukushima-ken). *Japan Analyst*, 1959, **8** (2), 128–129.—In HCl soln. containing Na citrate, a small amount of Fe (≤1 mg) is reduced with an excess of 0·01 *N* $TiCl_3$ and the excess of Ti is titrated with 0·01 *M* methylene blue (**I**) (Zn-free). For <1 mg of Fe, it is better to titrate **I** with $TiCl_3$. The time taken for a determination is ≃10 min. *Procedure*—Mix the sample (50 to 100 ml) with 20% Na citrate soln. (20 ml), pass a stream of CO_2 for 2 min. and warm to 90°; add 0·01 *N* $TiCl_3$ (20 ml) and after 2 min. titrate with 0·01 *M* **I**. For <1 mg of Fe, heat conc. HCl (5 ml), 20% Na citrate soln. (20 ml) and water (30 ml) to 90° in a stream of CO_2; add 0·01 *N* $TiCl_3$ (5 ml) and **I** soln. until a faint blue colour appears. Add the sample (10 ml) and titrate with 0·01 *N* $TiCl_3$ until the colour is discharged.

K. Saito

1005. **Determination of boron in iron alloys.** L. S. Evlashin and L. A. Zatuchnaya (Kirov Chelyabinsk Works). *Zavod. Lab.*, 1959, **25** (5), 580–581.—For the spark spectrographic determination of low contents (≃0·001%) of B in steel, the line pair B 2065·8 - Fe 2063·7 A is recommended.

G. S. Smith

1006. **Spectrochemical analysis of steel by transfer to counter-electrodes.** A. Garrido de Bartolomé. *Rev. Cienc. Apl.*, 1959, **13** (4), 322–325.—Chromium and Ni (>5%) are determined by volatilisation from an electrode of the sample on to a counter-electrode by means of an electric discharge. The counter-electrode may be of aluminium, zinc, carbon, silver or copper, copper giving the best results. The lower steel electrode is then replaced by one of the same material as the counter-electrode, and the spark discharge is photographed with a Hilger high-dispersion spectrograph and an exposure time of 1 min., a spark-gap of 1·5 mm and a Feussner spark generator being used. The time of deposition on the counter-electrode is 1 min. for Cr and 2 min. for Ni; the line pairs Fe II 2714·4 A - Cr II 2860·9 A, and Fe II 2883·7 A - Ni II 2610·09 A were used. By this procedure the density of the spectral lines, measured with a Zeiss microphotometer, is a linear function of the concn., and the continuous background is considerably reduced. In the analysis of steels containing 11, 17 or 27% of Cr, and 12, 17 or 33% of Ni, the coefficients of variation were ±8% (Cr) and ±10% (Ni). A comparison of the present procedure with that of direct registration of the spark spectrum between the sample and a copper electrode showed that the latter method gave no useful curves and suffered from interference by high concn. of other elements.

H. Friedmann

1007. **Determination of boron in plain-carbon and alloy steels.** J. Borrowdale, R. H. Jenkins and C. E. A. Shanahan (Central Res. Lab., Richard Thomas & Baldwins Ltd., Whitchurch, Aylesbury, Bucks., England). *Analyst*, 1959, **84**, 426–433.—The sample is dissolved in 20% v/v H_2SO_4 (20 ml) in a boiling-tube fitted with an air-condenser. The soln. is filtered and the insol. residue is washed with 15 ml of water. A little Na_2CO_3 is sprinkled on the residue on the filter-paper, which is then ignited at low temp., the remainder of 1 g of Na_2CO_3 is added and the mixture is fused at 1000°. The melt is dissolved in 20% H_2SO_4 and the soln. is combined with the original filtrate, which is then diluted to 50 ml. A 1-ml aliquot is distilled with methanol (40 ml), the distillate being collected beneath the surface of 5 ml of 0·1 *N* NaOH in a platinum dish, and all but 1 ml of the liquid being distilled. The methanol is removed by evaporation, and an oxalic acid soln. (containing HCl) and 5 ml of curcumin soln. (0·1 ml in 400 ml of ethanol) are added to the residue. The liquid is evaporated at 55°, the residue is extracted with aq. acetone (1:1), the extract is diluted to 50 ml with the same solvent and its extinction is measured against aq. acetone in an absorptiometer with Ilford No. 605 filters, corrected for a reagent blank and referred to a calibration graph prepared from soln. of B-free steel to which standard amounts of H_3BO_3 have been added.

A. O. Jones

1008. **Determination of nitrides of aluminium, silicon, vanadium and chromium in carbon steel and low-alloy steel.** E. Wrzesińska. *Prace Inst. Minist. Hutn.*, 1958, **10**, 180–184.—Beeghly's method for the determination of aluminium nitride (*Anal. Chem.*, 1949, **21**, 1513), in which the iron is dissolved by refluxing with Br in anhyd. methyl acetate and the insoluble nitrides recovered by filtration, can be applied to construction steel containing ≃0·4% of C and 1% of Cr, and to low-carbon steel containing 0·6 to 1·5% of Si and correspondingly 1·5 to 0·6% of Mn. The acid-insol. CrN, formed in the construction steel, can be calculated by the difference between the total N and the acid-sol. N in the insoluble fraction only in the absence of Al. The Si_3N_4 formed in the low-alloy of variable Mn and Si concn. can also be determined by the difference between the total N and the acid-sol. N. The determination of total N by the vacuum melting method is not necessary. It can be determined by digesting the insoluble fraction in conc. H_2SO_4 with the addition of K_2SO_4, followed by a Kjeldahl distillation.

Chem. Abstr.

1009. **Simultaneous spectrographic determination of carbon and alloying elements in steel.** A. S. Dem'yanchuk (E. O. Paton Inst. of Electro-welding, Acad. Sci., UkrSSR). *Zavod. Lab.*, 1959, **25** (5), 581–583.—The analysis is carried out with a high-frequency spark discharge between the sample and a magnesium counter-electrode (1·6 or 2·5 mm in diameter) and the use of two plates, one for photographing the carbon line 2296·89 A, which, for low contents of C (0·02 to 0·50%), is compared with Fe 2304·73 A, and for high contents (0·50 to 2·00%) is compared with Fe 2279·92 A. No pre-sparking is necessary. The contents of the alloying elements are found from measurements made on the other plate.

G. S. Smith

1010. **Determination of tin in iron and alloy steels.** A. I. Williams (National Physical Lab., Teddington, Middx., England). *Analyst*, 1959, **84**, 433–436.—The sample irradiated at pile factor 10 (10^{12} neutrons per sq. cm per sec.) for 6 hr. and allowed to decay for 24 hr. is dissolved in a mixture of 10 ml of 5 *N* HCl and 2 ml of a carrier soln. of Sn (10 mg per ml) at a temp. ⩾65°. The soln. is oxidised with bromine water, aq. NH_3 is added until the ppt. formed only just re-dissolves, and the soln. is saturated with H_2S, then centrifuged; the supernatant liquid is discarded and dissolution of the ppt. in HCl, oxidation with bromine and pptn. with H_2S are repeated twice. The ppt. is dissolved in HCl (1 ml), a slight excess of bromine water is added, scavenging is carried out with carrier soln. of Cu, Sb, Mo, La and Fe, and finally the Sn is pptd. as the sulphide and ignited to the oxide, weighed and counted. The activity is converted to weight of Sn by comparison with a standard sample of pure tin irradiated and treated in the same manner as the sample. Results agreed with those of a chemical method.

A. O. Jones

1011. **Application of tetraphenylarsonium chloride to the determination of tin in iron and steel.** Masayoshi Ishibashi, Taitiro Fujinaga and Masanori Sato (Chem. Dept., Fac. of Sci., Kyoto Univ., Sakyo-ku). *Japan Analyst*, 1959, **8** (2), 77–80.—The polarography of Sn^{IV} in the presence of tetraphenylarsonium chloride (**I**) (Kolthoff and Johnson, *Anal. Chem.*, 1951, **23**, 574) was examined. For the separation of Sn from interfering ions (including As and Cu), Sn is co-pptd. with MnO_2. *Procedure*—Dissolve the sample (1 g) in HNO_3 (1:2) (60 ml); boil the soln. with water (30 ml) and $MnSO_4$ soln. (20%) (4 ml) and add $KMnO_4$ soln. (3%) (2 ml) drop by drop. Filter off the MnO_2, and dissolve it in HCl (3 *N*) (5 ml) and H_2O_2 (30%, a few drops); evaporate the soln. to 2 ml, and add ascorbic acid (1 g), conc. HCl, **I** and ethanol to produce 20 ml of a supporting electrolyte containing 3·5 *N* HCl, 5×10^{-4} *M* **I** and 10% of ethanol.

K. Saito

1012. **New colorimetric methods for the determination of phosphorus in carbon steel and cast iron.** R. Aragonés Apodaca. *Quím. e Ind., Bilbao*, 1959, **6** (2), 45–47.—The sample is digested with $HClO_4$ - H_2SO_4 - H_2O (6:1:3), and Fe^{III} is reduced to Fe^{II} with $NaHSO_3$. The P is then determined colorimetrically with the ammonium molybdate reagent of Lucena-Conde and Prat (*cf. Anal. Abstr.*, 1957, **4**, 3911). Satisfactory results are obtained with P contents up to 0·1%. Interfering elements include W, As, Ge, Cr and V, and others (Al, Co, Cu, Ni, Mn and Sn^{II}) not normally present in steel in sufficient proportion to cause difficulty.

L. A. O'Neill

1013. **Spectrophotometric determination of small contents of niobium and tantalum in stainless steel.** R. Rosotte and E. Jaudon (IRSID, Saint-Germain-en-Laye, France). *Chim. Anal.*, 1959, **41** (6), 229–239.—Niobium from European sources frequently contains Ta at a level of ≃10%. Stainless steel containing 0·5% of Nb therefore contains ≃0·05% of Ta, and a method is proposed which is applicable to this level. After pptn. and filtration of the hydrated oxides of Ta, Nb and some Ti, the oxides are ignited, and dissolved in H_2SO_4. Tantalum is then determined with catechol at 395 mμ, Nb with NaSCN at 385 mμ after extraction into ethyl acetate, and Ti with chromotropic acid at 460 mμ. Each determination has been studied to establish the optimum conditions and the mutual influence of the several reagents. In particular, the interference of Ti in the determination of Ta and Nb has

been established and the necessary correction is given. A 2·5-g sample is used (5 g for Nb <0·3%). Recoveries on synthetic standards (6) are within 2% of the amount added (0·020% of Ta, 0·20% to 0·50% of Nb). T. R. ANDREW

1014. **The analysis of oxide inclusions in steel in the microgram range. II. The determination of silica, alumina, iron oxide and manganese oxide.** S. Meyer and O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen/Saar, Germany). *Mikrochim. Acta*, 1959, (5), 720–724 (in German).—The method is an improvement on that previously described (*Ibid.*, 1958, 744). The sample (200 to 400 μg) is fumed with HF to determine SiO_2 as the loss in weight; the residue is fused with Na_2CO_3 - K_2CO_3 - borax and Fe is extracted with diethyl ether and measured photometrically with 1:10-phenanthroline; Mn is then extracted with Na diethyldithiocarbamate - $CHCl_3$ and determined photometrically; and Al is extracted with 8-hydroxyquinoline and determined photometrically as hydroxyquinolinate. A batch of four samples may be completed in 5·5 to 7·5 hr. The relative error is ±5 to 10%. D. F. PHILLIPS

1015. **Determination of sulphur and phosphorus in iron alloys with exchange resins.** G. Venturello and C. Gualandi (Ist. Chim. "C. Ciamician," Univ. Bologna, Italy). *Boll. Sci. Fac. Chim. Bologna*, 1959, **17** (1), 5–8.—*Procedure*—The sample (3 to 5 g) is dissolved by boiling with HNO_3 (75 ml) and H_2O (50 ml), and the soln. is evaporated to dryness 4 times with 25 ml of conc. HCl. The residue is treated with H_2O (300 ml), any undissolved material is filtered off, and the filtrate is concentrated to 200 ml. Solid NaOH is added until $Fe(OH)_3$ begins to precipitate, then the soln. is acidified with HCl, to a total vol. of 300 to 500 ml, and passed through a column of anion-exchange resin (IRA-410) (480 to 490 mesh) at a rate of 20 to 30 drops per min., and the column is washed with 250 to 300 ml of H_2O. For the determination of SO_4^{2-} and PO_4^{3-}, the column is eluted successively with 100 ml of 0·5 *N* NaOH, with H_2O till neutral, with 100 ml of 0·5 *N* HCl, and finally with H_2O, repeating the alkaline and acid elutions twice. The SO_4^{2-} are determined gravimetrically as $BaSO_4$ and the PO_4^{3-} are determined in the filtrate as ammonium molybdophosphate after making it alkaline with aq. NH_3. The mean error is <1% in both determinations. L. ZANONI

1016. **Neutron activation analysis methods for the group-VIII elements.** C. E. Miller (Oak Ridge Nat. Lab., Tenn., U.S.A.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2715, 1959, 113 pp.—The conclusions that are reached from this investigation of short-lived radionuclides of the group-VIII elements are that ^{60m}Co, ^{104m}Rh, ^{109m}Pd and ^{193}Pt can all be used to good advantage. With ^{109m}Pd the limit of quant. measurement will be higher by the use of the short-lived radionuclide than by the use of the long-lived radionuclide. However, the short duration of irradiation provides some advantage in the length of time required to perform a determination of Pd. It is also shown that Os has no short-lived radionuclide that can be used as a means of analysis. It has been shown that the short-lived radionuclide of Ir does not produce radioactive interference in the determination by activation analysis of the platinum-group metals. A 0·097-MeV γ-ray has been found in the γ-ray spectrum of ^{104}Rh. This γ-ray has not previously been accounted for in the decay scheme of ^{104}Rh. NUCL. SCI. ABSTR.

1017. **Azo dyes as reagents for cobalt.** I. M. Korenman, N. V. Kurina and A. A. Karnishin. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 377–382; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,556.—Of 68 azo dyes studied, 3 give reactions with cobalt salts; the sensitivity of these reactions is determined. Salts of Fe and Cu interfere in the detection of Co; Mn, Cr and Al do not. The limiting ratios of the various elements in the detection of Co are found. The reactions of Co with Diamond red PV (**I**) and Acid Chrome blue black K (**II**) are used for the photometric determination of Co. To 3 ml of the test soln., containing 0·5 to 10 μg of Co^{2+}, add 0·6 ml of a 0·1% soln. of **I** or **II**, 3 ml of a 3·5% soln. of $NaNO_2$, 3 ml of 0·5 *N* acetic acid, and water to 10 ml; set aside for 10 min., and measure the extinction in 10-mm cells at 580 mμ (with **I**) or at 540 mμ (with **II**). The reaction with **I** can be used to determine 0·5 to 8 μg of Co, and that with **II** for 1 to 9 μg of Co in 3 ml of soln. C. D. KOPKIN

1018. **Determination of small amounts of cobalt with 2-nitroso-1-naphthol by extraction with chloroform.** W. Nielsch (Karl-Reiss-Platz 12, Mannheim, Germany). *Mikrochim. Acta*, 1959, (5), 725–734 (in German).—Cobalt in concn. <0·12 μg per ml can be quantitatively pptd. by 2-nitroso-1-naphthol and subsequently extracted with $CHCl_3$. The usual waiting period of 24 hr. for the completion of the pptn. may be reduced to 30 min. by vigorous shaking before the addition of the $CHCl_3$. The cobalt complex is pptd. in the pH range 2 to 9 and, after a single extraction and purification of the $CHCl_3$ phase with NaOH and HCl, may be directly measured by photometry. D. F. PHILLIPS

1019. **Macro- and micro-determination of traces of cobalt. I. Cathode-ray polarographic study of cobalt.** D. Monnier, J. Vogel, W. Haerdi and P. E. Wenger (Lab. de Chim. Minérale, Univ., Genève, Switzerland). *Helv. Chim. Acta*, 1959, **42** (5), 1672–1678 (in French).—Concentrations of 0·1 μg of Co per ml in volumes of 0·1 ml have been measured by using a dropping mercury cathode, a mercury-pool anode and 0·1 *M* KCl as electrolyte in an atmosphere of N. Macro- and micro-cells for the work are illustrated. The standard deviations and 95% confidence limits for the various cells are given. E. G. CUMMINS

1020. **Amperometric determination of cobalt by an iodimetric method with a rotating platinum micro-electrode.** A. K. Zhdanov, V. A. Khadeev and Ya. G. Yakovenko (Lenin Mid-Asia Univ., Tashkent, USSR). *Zhur. Anal. Khim.*, 1959, **14** (3), 367–369.—*Procedure*—Add NH_4NO_3 (25 g) to the cobalt soln., warm to room temp., and add iodine soln. in excess to obtain a subsequent titre of ≮1 to 2 ml. Add 18% aq. NH_3 soln. (5 ml) and dilute to 40 to 50 ml. After 5 to 10 min. titrate the excess of iodine amperometrically with sodium arsenite soln. with an applied potential of +0·1 V. Ions of Ca, Sr, Ba, Zn, Mg, Al, Fe^{III}, Ni, Cd, Cu, Pb and Bi do not interfere even in large amounts. Concn. of Co above 0·01 mg per ml can be determined satisfactorily. K. R. C.

1021. **Analysis of trace activities of cobalt-60 in Hanford-treated waste solutions.** H. R. Helmholz and R. A. Schneider (Gen. Electric Co., Richland, Wash., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1151–1157.—For the determination of ^{60}Co (present as a stable cobaltic ammine or as the cobalticyanide complex, or both, in irradiated uranium processing soln.), the Co is pptd. as a mixture of cobaltous and

ferrous cobalticyanides after the destruction of the cobaltic ammine complexes by reduction with hydroxylamine and then heating with acid. Aquopenta-amminocobaltic nitrate is used to indicate visually that the reactions are taking place. The ppt. (containing >99·9% of the Co) is dissolved in fuming H_2SO_4 plus KIO_3, the Ru being volatilised by continuous heating. The soln. is then scavenged with $Fe(OH)_3$, aq. NH_3 being used to complex Co^{2+}; CoS is pptd. from the filtrate, washed with dil. HCl and dissolved in HNO_3. The specific activity of ^{60}Co is determined on dry $Co(NO_3)_2$ by a combination of γ-energy discrimination and γ-coincidence counting. Full details are given of the instrumentation and procedures for counting. The mean radiochemical yield of ^{60}Co is 98 ± 1%, and the standard deviation of counting is ±6%. The separation factor for the other radionuclides is 10^5. A special decontamination step is necessary when the sample is high in ^{137}Cs. W. J. Baker

1022. **Separation and colorimetric determination of cobalt in presence of copper and nickel.** W. D. Duffield (26, Park Crescent, Portland Place, London). *Analyst*, 1959, **84**, 455–457.—The sample soln. is adjusted to pH 8 with an aq. NH_3 - citric acid mixture and extracted repeatedly with a 10% soln. of 8-hydroxyquinoline in $CHCl_3$ until the extract is colourless. The extract is evaporated to dryness, the residue is heated to dryness with a little aqua regia and dissolved in 0·1 to 0·2 ml of 50% HCl. The whole of the soln. is applied to a guide line on a sheet of chromatographic paper and ascending chromatography is carried out with ethyl methyl ketone - conc. HCl - water (15:3:2) as solvent. The air-dried paper is neutralised in NH_3 vapour and then sprayed with dithio-oxamide soln. [1% in ethanol - water (3:2)]. The zone due to Co appears as a yellow band midway between the upper band due to Cu (olive green) and the lower band due to Ni (blue). The appropriate section is cut out and ignited at 550°, the ash is treated with aqua regia and dissolved in dil. HCl, and the Co is determined colorimetrically with nitroso-R salt. If the amount of each metal present is <2 μg, a parallel tracer strip charged with this amount of each metal may be incorporated in the paper and separated from the main chromatogram by a slot. Recovery of known amounts of Co was good.
A. O. Jones

1023. **New rapid gravimetric method for the semi-micro determination of cobalt and nickel as diliturate.** J. Dick and F. Mihai (Inst. Politeh., Timişoara, Romania). *Acad. R.P.R., Baza Cercet. Ştiinţ. Timişoara, Stud. Cercet. Ştiinţ., Ser. I*, 1958, **5**, 107–111.—The method depends on the pptn. and direct weighing of the cobalt or nickel diliturate, $Co(C_4H_2O_3N_2NO_3)_2.8H_2O$ or $Ni(C_4H_2O_3N_2NO_3)_2.8H_2O$. This is a highly selective method; the determination can be made in 15 min. and is very accurate.
Nucl. Sci. Abstr.

1024. **Photometric method for the determination of nickel.** M. Ya. Shapiro (Pirogov Med. Inst., Odessa). *Zhur. Anal. Khim.*, 1959, **14** (3), 365–366.—An aq. soln. of murexide reacts with an ammoniacal complex of Ni to give an orange - yellow colour, which obey's Beer's law. It is possible to detect 0·2 μg of Ni in 1 ml of soln. *Procedure for the determination of Ni in steel*—Dissolve the sample (0·10 to 0·20 g) by boiling with H_2SO_4 (1:9), add H_2O_2 to oxidise Fe^{2+} to Fe^{3+}, and neutralise the soln. with aq. NH_3 soln. until the Fe begins to precipitate (pH 6·2 to 6·4). Add 0·5 *N* $ZnSO_4$ (10 ml), stir, add *N* $K_4Fe(CN)_6$ (10 ml), filter off the $Zn_3[Fe(CN)_6]_2$ ppt. containing the Ni and wash several times with H_2O. Pour an excess of 5% NaOH soln. satd. with $Zn_3[Fe(CN)_6]_2$ through the filter; the hydroxides of Fe and Ni are left on the filter; dissolve this residue in H_2SO_4 (1:9), neutralise with an excess of aq. NH_3 soln., and boil to remove the excess. Dilute the soln. to 100 ml and take a 10-ml aliquot for photometric determination with 0·02% aq. murexide soln. With 0·10% of Ni in 0·1 g of steel 100% recovery was obtained; the recovery tends to diminish with increasing concn. of Ni.
K. R. C.

1025. **Determinations in inorganic analysis by extraction with the use of coloured cations. II. Use of complexes of 1:10-phenanthroline for the separation and determination of small quantities of nickel in the presence of iron and cobalt.** L. Ducret (Éc. Nat. des Ind. Agric. et Aliment., Paris, France). *Anal. Chim. Acta*, 1959, **20** (6), 568–572 (in French).—The oxidised forms of the 1:10-phenanthroline complexes of Fe and Co are not extracted from aq. soln. by 1:2-dichloroethane, but the complex of Ni passes into the organic layer and is determined spectrophotometrically. The soln. for analysis must contain ≯16 mg of alloy, of which ≯5 mg is Fe, in 5 ml of dil. H_2SO_4 or dil. HCl of pH ≃1. *Procedure*—To the alloy soln. add 0·5 ml of 5% aq. $K_2S_2O_5$ soln. and, after 1 min., 10 ml of 1:10-phenanthroline sulphate soln. (≃0·1 *M*) (**I**). Set aside for 5 min. and then add 0·1 *N* $Ce(SO_4)_2$ (**II**) until the Fe complex turns blue. Add a further 2 ml of **II** and 3 ml of 0·5 *M* trichloroacetic acid (**III**). Shake the soln. for 3 min. with 10 ml of 1:2-dichloroethane and separate. Shake the organic layer with a mixture of 10 ml of 0·01 *M* $Ce(SO_4)_2$ in 0·2 *N* H_2SO_4. 1 ml of **I** and 1 ml of **III**. Separate and centrifuge the organic layer and measure its extinction at 520 mμ in a 2-cm cell. Prepare a calibration graph by the same method. At the 10-mg level the relative error is ≃1%.
H. N. S.

1026. **Complexometric determination of nickel and iron in the same sample by photometric titration.** A. Dewald. *Acad. R P.R., Baza Cercet. Ştiinţ. Timişoara, Stud. Cercet. Ştiinţ., Ser. I*, 1958, **5**, 125–129.—The iron is masked with pyrophosphate, and the Ni is titrated directly with EDTA in the presence of murexide up to the violet colour change. An IF620 filter is used. The iron is then titrated indirectly; an excess of EDTA is added, which destroys the pyrophosphate complex, and the excess is titrated with a standard soln. of $Ni(NO_3)_2$, with an IF465 filter.
Nucl. Sci. Abstr.

1027. **Colorimetric determination of palladium with 1-phenylthiosemicarbazide.** Sumio Komatsu and Kazuo Taki (Fac. of Lit. and Sci., Toyama Univ.). *J. Chem. Soc. Japan, Pure Chem. Sect.*, 1959, **80** (4), 397–398.—The complex of Pd with 1-phenylthiosemicarbazide (**I**) dissolves in an excess of **I** to give an orange - yellow soln. (max. absorption at 372 mμ), which remains unchanged for 1·5 hr. The calibration curve is linear for <25 μg of Pd per ml in acetic acid (>3 *N*) containing a molar ratio of **I** to Pd >13. The error is ≃2% for >1 μg of Pd per ml. Ferric iron is masked with NaF, and Cu and Hg with EDTA. Tervalent Au is removed by extraction with ether. Quadrivalent Pt produces a green ppt. with **I**; the pptn. is quant. after 3 hr. in 3 *N* acetic acid. The Pt - **I** complex is filtered off and the Pd determined in the filtrate.
K. Saito

1028. **Estimation of palladium by αβ-dioximinoacetoacetanilide** [**αβ-dioximinobutyranilide.**] J. S. Dave and A. M. Talati (M.S. Univ., Baroda). *J. Indian Chem. Soc.*, 1959, **36** (3), 179–182.—Palladium is pptd. quant. by αβ-dioximinobutyranilide as $Pd(C_{10}H_{10}O_2N_3)_2$ from fairly acid soln. (pH 0·4) and the ppt. is dried at 110° to 120° to constant wt. Under the experimental conditions, no other metal complex of the reagent is formed; Hg, Sn, Co, Ni, Zn, Cd, Cu, Mn, Ca, Ba, Mg, Fe^{III} and Cr do not interfere; no ppt. was obtained in the presence of KI, but the presence of KCN caused low results and $NaNO_3$ caused high ones. Sodium acetate, tartaric acid and K_2SO_4 do not interfere in acid soln., nor do KBr or KI in neutral or alkaline soln.

O. M. WHITTON

1029. **Spectrophotometric determination of palladium(II) with tin(II).** G. H. Ayres and J. H. Alsop, III (Univ. of Texas, Austin, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1135–1138.—From 8 to 32 p.p.m. of Pd^{2+} can be determined by adding to the sample soln. ($PdCl_2$) 10 ml of mixed acid (2·4 *M* in HCl and 2·3 *M* in $HClO_4$), followed by 2 ml of 0·5 *M* $SnCl_2$ in 1 *M* HCl and, after dilution to 25 ml and setting aside for 30 min., measuring the extinction of the green complex at 635 mμ in a 1-cm cell. Lower concn. (0·5 to 2·5 p.p.m.) can be determined by adding to a Cl^--free sample soln. dil. $HClO_4$ and dil. H_3PO_4, followed by 2 ml of stannous phosphate soln. (0·0065 *M* in Sn^{2+} and 1·6 *M* in H_3PO_4) and, after dilution to 25 ml (the soln. should then be 1·16 *M* in $HClO_4$ and 0·77 *M* in H_3PO_4), measuring the extinction of the deep-red complex at 487 mμ in a 1-cm cell. The colour develops within 10 min. and is stable for 90 min. The error is within ≃0·6%. The effects of variables and the tolerances for foreign ions are discussed for each procedure. The stannous phosphate method has a higher sensitivity than most of the usual spectrophotometric methods for Pd. W. J. BAKER

1030. **Colorimetric determination of palladium(II) in the presence of platinum(IV).** G. Popa, D. Negoiu and G. Baiulescu (Fac. de Chim., Univ. "C. I. Parhon," Bucureşti, Romania). *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **7** (1), 73–78.—From a consideration of the theory of the functional and active analytical groups attached to the organic molecule, two new reagents, Tropaeolin OO (C.I. Acid Orange 5) (**I**) and Tropaeolin O (C.I. Acid Orange 6) (**II**), are proposed for this determination. With **I**, 1 ml of Na acetate soln. (2%) plus 4 ml of an ethanolic soln. of **I** (0·01%) is added to both test soln. and control, which are then diluted to 10 ml with ethanol. A reading is taken immediately in a Pulfrich photometer at 496 mμ. From 2·45 to 39·3 μg of Pd per ml has been determined with the concn. of Pt not more than double that of Pd. With **II**, 2 ml of an aq. soln. (0·05%) is used with the test soln. and diluted to 10 ml with water. The reading is taken immediately at 533 mμ. From 0·63 to 9·83 of Pd per ml has been determined in the presence of 240 times the amount of Pt.

H. SHER

1031. **Spectrophotometric determination of osmium. I. Anthranilic acid as a reagent.** A. K. Majumdar and J. G. Sen Gupta (Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **20** (6), 532–539.—Anthranilic acid yields a violet complex (absorption max. at 460 mμ) with Os^{4+}, Os^{6+} and Os^{8+}. A 1:1 complex with Os^{4+} is formed, the higher valency states being reduced to Os^{4+} before complex formation. The optimum concn. range for the absorptiometric determination of Os is 2 to 6 p.p.m. An acetate buffer, pH 5·9, is added to keep the pH within the optimum range (5·5 to 6·5), and the max. colour is developed with Os^{4+} by heating the reactants on a water bath for 10 min. With Os^{6+} and Os^{8+} the max. colour is developed in 1 hr. at room temp. The accuracy and sensitivity of the method and the dissociation constant of the complex are determined. Many metal ions interfere, but some of them can be masked with EDTA. Copper, Cd, Al, Th, Zr, Sb, Bi and rare-earth metals must be absent. Gold and Pt interfere. H. N. S.

1032. **Determination of platinum and palladium in ores and concentrates. New fire-assay method.** M. E. V. Plummer and F. E. Beamish (Univ., Toronto, Ont., Canada). *Anal. Chem.*, 1959, **31** (7), 1141–1143.—Microgram amounts of Pt and Pd (= 0·02 to 0·03 oz of each per ton) can be collected by an Fe - Cu - Ni alloy in a clay crucible from a fused mixture of the roasted sample, Na_2CO_3, borax and graphite. A standard air - gas furnace (1050° to 1485°) is used. The recovery (Pt 99·3%, Pd 98·2%) is equal to that of the usual fire-assay method, and neither Ni nor other metals of the Pt group interfere. The new method ensures complete transfer of Ni to the button (20 g), besides ensuring the absence of Pt and Pd from the slag (unless the ore is very high in Ni). The button is dissolved in conc. HCl and the dil. soln., after treatment and removal of SiO_2, is passed through a short column of Dowex 50-X8 (free from C, B and Se), and the Pt and Pd in the eluate are determined by standard colorimetric procedures. W. J. BAKER

1033. **Quantitative X-ray spectrographic determination of platinum in ores.** N. F. Losev. *Sb. Nauch. Trud. Irkutsk. Nauch.-Issled. Inst. Redk. Met.*, 1958, (7), 25–28; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,563.—A Cauchois spectrometer is used, with a quartz crystal analyser cut along a surface of the prism (edge 1010) and curved to a radius of 700 mm; Ta is used as comparison element. The analytical lines are Pt $L\alpha_1$, Ta $L\beta_1$ and Ta $L\beta_2$. To prepare the sample, dissolve the assay button in HNO_3 (1:2), adding the required amount of Ta_2O_5, evaporate the soln., ignite the residue and carefully grind it. With a concn. of Pt < 0·5%, the button is first enriched by dissolving all or part of its silver content with H_2SO_4 (1:9) and the enriched product is treated as described above. The intensity coeff. is determined from standard samples. The error is 3 to 4%. C. D. KOPKIN

1034. **Determination of the mineral composition of carbonate rocks.** B. V. Ivanovskii and I. N. Volodina. *Zap. Leningr. Gorn. Inst.*, 1958, **33** (2), 236–242; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,589.—A volumetric method is described for the phase analysis of carbonate rocks, which permits the separate determination of the content of calcite, dolomite and magnesite. The method is based on the differing rates of reaction of $CaCO_3$ and $CaMg(CO_3)_2$ with HCl in the cold, and the property of $MgCO_3$ of reacting with HCl only on warming. The form of the curves defining the vol. of liberated CO_2 as a function of time gives a qual. picture of the phase composition of the rocks. The method gives the approximate amounts of the separate phases. Results are given of the volumetric analysis of 8 samples of carbonate rock, which agree with the chemical and mineralogical analyses. It is concluded that the volumetric method gives a more accurate picture of the phase composition of rocks than the mineralogical method. C. D. KOPKIN

1035. **Complexometric determination of the sum of titanium and aluminium in clay.** E. A. Nechaeva and E. S. Lapidus (Petrovskii Dniepropetrovsk Metallurgical Works). *Zavod. Lab.*, 1959, **25** (5), 544–545.—A rapid method, which avoids the need for separating SiO_2 and is suitable in the presence of Fe and the alkaline-earth metals, is described. The sample (0·5 g) is fused with Na_2CO_3, the melt is dissolved in dil. HCl, and the soln. is diluted to 250 ml. An aliquot (50 ml) is mixed with 20 ml of 0·05 *M* EDTA (disodium salt), then neutralised to methyl red indicator with aq. NH_3 and treated with 20 ml of a buffer soln. (prepared by adding 20 ml of glacial acetic acid to a soln. of 500 g of ammonium acetate in 1 litre of water). The soln. is boiled for 3 min., then cooled (20° to 25°) and treated with 1 ml of 0·1% $K_4Fe(CN)_6$ soln., 1 ml of 0·2% $K_3Fe(CN)_6$ soln. and 1 ml of a 1% soln. of benzidine in glacial acetic acid, and titrated to the appearance of a blue colour with 0·05 *M* Zn acetate. The titrated soln. is boiled with 30 ml of saturated NaF soln., thus decomposing the complexes of Al and Ti. The cooled soln. is treated with the indicator soln. as before, and the liberated EDTA is titrated with 0·05 *M* Zn acetate. G. S. SMITH

1036. **Spectrographic analysis of mica.** V. V. Plastinin (A. A. Zhdanov Irkutsk State Univ.). *Zavod. Lab.*, 1959, **25** (5), 577–580.—An a.c. arc method for determining Fe, Si, Al, Mg, Mn and Ca in mica, and a spark method, requiring fusion of the sample, for more accurate determinations of high contents are described. For the arc method, the sample (150 mesh) is mixed with an equal weight of graphite powder containing 1% of Co as $CoSO_4.7H_2O$ (to serve as internal standard) and 0·03% of V as V_2O_5 (to give the arc temp. from measurements of relative line intensities) and completely evaporated (≃4 min.) from a carbon electrode in an arc (16 amp.). Standards to simulate the composition range of samples of mica are prepared from the oxides (the SiO_2 used being naturally occurring quartz) in a base mixture of K_2CO_3, Na_2CO_3 and BaO (3:1:1). The line pairs used are Fe 2447·71 - Co 2441·05, Al 3050·08 - Co 3048·89, Si 2452·14 - Co 2441·05, Mg 3336·68 - Co 3409·18, Mn 2595·76 - Co 2441·05 and Ca 3179·33 - Co 3147·06 A. For the spark method, the samples (or standards) (0·5 g) are mixed with $CoSO_4.7H_2O$ (0·6 g) (internal standard) and borax (5·0 g) and fused at 900°, and the cooled melt is powdered, mixed with carbon (1:1) and briquetted at 1500 kg per sq. cm. The line pairs used are Si 2516·12 - Co 2521·36, Al 3082·15 - Co 3072·34, Mn 2801·06 - Co 2648·64, Fe 2488·15 - Co 2521·36, Mg 2776·69 - Co 2648·64, and Ca 3179·33 - Co 3072·34 A.
G. S. SMITH

1037. **Determination of quartz and opal in rocks.** V. P. Astaf'ev. Symposium: Determination of free silica in rocks and ore dust. Moscow. *Akad. Nauk SSSR*, 1958, 51–53; *Ref. Zhur., Khim.*, 1959, (10), Abstr. No. 34,630.—The method described is based on the insolubility of quartz and opal in fused $NaHSO_4$. Ignite the sample (1 g) (80 to 100 mesh) for 1 hr. at 700°, cool, dissolve in 15 ml of conc. HCl with gentle boiling (≃30 min.), and dilute with hot water (30 to 40 ml). Filter off the insol. residue and wash it with 1% HCl (×3) and with water (×4). Carefully ash the filter and residue, cool, and fuse for 1 hr. with 6 or 8 times the amount of anhyd. $NaHSO_4$ at 400° to 550°, periodically cooling the melt and adding small portions of $NaHSO_4$. Extract the melt with water, filter off the insol. residue, wash it with hot water till free from SO_4^{2-}, and ignite. To separate quartz and opal from combined silica, treat the washed and ignited residue with 10% NaOH soln. (50 ml) while heating to 80° to 100°; filter, wash the residue with 1% HCl till neutral to phenolphthalein, dry, ignite at 1000° to 1200°, and weigh. Then add a few drops of conc. H_2SO_4 and 3 to 5 ml of HF, evaporate on a sand bath to remove fumes of SO_3, ignite the residue at 900° to 1000°, again weigh, and calculate the content of quartz and opal from the decrease in weight.
C. D. KOPKIN

1038. **Infra-red spectroscopic determination of quartz in pit dusts.** M. Gade and K. F. Luft (Bergbau-Forsch. G.m.b.H., Forschungsinst. d. Steinkohlenbergbauvereins, Essen). *Naturwissenschaften*, 1959, **46** (9), 315–316.—Quartz may be detected and determined in samples of powdered coal or mineral ores by its absorption bands at 12·5 μ, 12·8 μ and 14·4 μ. To reduce background interference, heat the sample at 700° before i.r. examination. The error is $\simeq \pm 1\%$ absolute.
A. R. ROGERS

1039. **Spectrographic determination of trace elements in wolframite.** K. Rossmanith and Z. G. Hanna (I Chem. Inst., Univ., Wien). *Monatsh. Chem.*, 1959, **90** (1), 76–85.—Details are given of a spectrographic procedure for the determination of traces of Co, Ni, Cu, V, Sn, Cr, Pb and Mo in wolframite; down to about 0·001% of these elements can be determined with a mean error of 4 to 10%, except for Cr and Mo for which the method is less sensitive. Neither As nor P can be determined satisfactorily. The lines used are (in A) Co I 3453·50, Ni I 3414·76, Cu I 3273·96, V I 3185·39, Sn I 3175·01, Cr II 2849·83, Mo I 3170·34 and Pb I 2614·17; Fe 3196·94 is used as a standard. N. E.

1040. **[Determination of] bismuth, thallium and mercury in stone meteorites by activation analysis.** W. D. Ehmann and J. R. Huizenga (Argonne Nat. Lab., Lemont, Ill.). *Geochim. et Cosmoch. Acta*, 1959, **17** (1–2), 125–135.—The slow neutron activation of meteorite samples is carried out in a heavy-water-moderated reactor, with a flux density of the order of 3×10^{13} neutrons per sq. cm per sec. for 10 to 15 days. Bismuth is determined by counting the 5·298-MeV α-particles of ^{210}Po (138·40 days), the active daughter of ^{210}Bi, a 5-day β-emitter produced by an (n, γ) process on 100% natural abundance ^{209}Bi; Tl is determined by β-counting (E_{max} = 0·765 MeV) the 4·0-year ^{204}Tl produced by an (n, γ) reaction on 29·50% natural abundance ^{203}Tl; Hg is determined by counting the 279-keV γ-ray associated with the decay of 45·8-day ^{203}Hg formed in the neutron irradiation by an (n, γ) reaction on 29·8% natural abundance ^{202}Hg. The radiochemical procedures are described in detail.
R. A. HOWIE

1041. **Determination of ignition loss in Portland blast-furnace-slag cements.** B. Chaiken (Bureau of Pub. Roads., Dept. of Commerce, Washington, D.C., U.S.A.). *Bull. A.S.T.M.*, 1959, (238), 53–58.—Owing to the oxidation of sulphides, the ignition losses of slag cements as normally determined are in error, and a correction assuming that all S^{2-} are oxidised to SO_4^{2-} is invalid. Two more accurate procedures are given—(*i*) a single ignition for 15 min., and the application of a correction for the observed increase in the content of SO_4^{2-} (revised

A.S.T.M. method), and (*ii*) ignition in an atmosphere of He at 900° to 1000° for 15 min., and thence for 5 min. to const. wt. The second method is more rapid and direct and is recommended for routine analyses of slag cements and slags.

W. J. BAKER

1042. **Complexometric determination of alumina in cement.** J. Calleja and J. M. Fernández Paris (Inst. Téc. de la Construcción y del Cemento, Madrid). *Rev. Cienc. Apl.*, 1959, **13** (4), 325–333.—To an aliquot of the filtrate (*e.g.*, 50 ml from 200 ml) from the conventional SiO_2 pptn. of 1 g of sample is added a known vol. (≃ 14 ml) of 0·05 *M* EDTA (disodium salt) (**I**), and conc. aq. NH_3 is added until the hydroxides of Fe and Al are pptd. Then the system is adjusted to pH 4·5 with 50 ml of buffer soln. (78 g of ammonium acetate and 60 ml of glacial acetic acid made up to 1 litre with water), boiled for 5 min., and cooled rapidly; abs. ethanol (60 ml) and dithizone soln. (0·025% in abs. ethanol) (3 ml) are added, giving a green soln., which is titrated wth 0·05 *M* Zn soln. (3·269 g of zinc dissolved in 8 ml of conc. HCl and made up to 1 litre) to a reddish end-point. The difference between the vol. of **I** soln. added and that of the Zn soln. used, multiplied by 2·549, gives the Al_2O_3 (mg). The Fe_2O_3 is determined in another aliquot by titration with 0·05 *M* $K_2Cr_2O_7$, with Ba diphenylaminesulphonate as indicator. When a high Fe content impairs the sharpness of the end-point in the titration with **I**, a higher dilution may be used without effect on the accuracy. Results for cement samples showed that the method has an accuracy equal to that of the gravimetric determination of Al_2O_3. The Ca and Mg present in cement do not interfere.

H. FRIEDMANN

1043. **Determination of the gypsum content of cement by means of cationite.** I. V. Bogdanova (All-Union Inst. of the Cement Industry for Sci. Res. and Design, Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (3), 373–374.—*Procedure*—Treat the sample (0·5 g) with H_2O (25 ml) stirring continuously for 10 min. Filter the soln. and wash the insol. residue (× 2 or 3) by decantation and then on the filter with 5% boric acid soln. (× 3 or 4) which dissolves the gypsum. Pass the combined filtrate and washings through a column of cationite (H form) at the rate of 3 to 4 ml per min., elute with H_2O (50 to 70 ml) at the same rate and titrate the combined eluates with NaOH soln. to methyl orange. The results do not differ by more than 0·15% (absolute) from those obtained gravimetrically.

K. R. C.

1044. **Identification of Lucas 700 headlamp-glass fragments by their physical properties.** D. F. Nelson (The Dominion Lab., P.O. Box No. 562, Dunedin, New Zealand). *Analyst*, 1959, **84**, 388–392.—The physical properties of fragments from 50 different Lucas headlamps were examined to ascertain their usefulness in the identification of fragments in forensic work. The density was determined by flotation in a bromoform - xylene mixture, adjustment being made by the addition of one or other component until the fragment was suspended. The density of the liquid was then determined. The refractive index was determined by observing the Becke line when the chips were immersed in liquids of known refractive index (*e.g.*, mixtures of castor and clove oils). Six arbitrary colour standards were selected from among the samples and the remainder were assigned to these groups. Arbitrary standards of hardness were chosen by determining their mutual scratching powers. Dispersion and fluorescence were found to be of little practical value. The results are tabulated and examined statistically. They can be used to calculate the probabilities that two glasses from different lamps would be indistinguishable in any particular instance.

A. O. JONES

See also Abstracts—**851**, Applications of Na tetraphenylboron. **854**, Indicators for titrations with ceric perchlorate. **855**, ICl_3 as volumetric reagent. **859**, Use of dielectric constants in analysis of rutile and anatase. **1192**, Glass still for fluoride analysis. **1193**, Teflon vessel for decomposition of rock samples. **1203**, Determination of P, S and Cl^- in graphite. **1209**, Internal standard method of spectrographic analysis. **1229**, Oxygen in metals. **1230**, Isotopic analysis of RbCl.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather, explosives.

1045. **Use of high-frequency methods in determining the composition of heterogeneous ternary liquid systems.** E. L. Heric (Univ. of Georgia, Athens, U.S.A.). *Chemist Analyst*, 1959, **48** (2), 31–33.—The high-frequency measurement of refractive index has been applied to a study of the system ethyl acetate - furfuraldehyde - water. The techniques used and the special precautions necessary to avoid the oxidation of furfuraldehyde (since the instrument is highly sensitive to acidic oxidation products) are described. The results for complete solubility curves and for the solubility of immiscible pairs in the system are compared with values from the literature. The agreement is satisfactory. High-frequency measurements eliminate any possible errors due to evaporation which might occur in conventional refractive-index measurements.

S. M. MARSH

1046. **Progress report on quantitative organic elementary micro-analysis. II.** W. Schöniger (Sandoz A.-G., Basel, Switzerland). *Mikrochim. Acta*, 1959, (5), 670–692 (in German).—A review is presented of methods for the determination of C and H, O, N, halogens, S, P and metals in organic materials, published since the author's last review (*Ibid.*, 1956, 1456), including developments in balances, weighing techniques and general laboratory procedures. (262 references.)

D. F. PHILLIPS

1047. **Carbon and hydrogen micro-determination by automatic combustion control.** E. Stehr (Texaco Res. Center, The Texas Co., Beacon, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1274–1278.—The rate of combustion in the micro-analytical apparatus described is accurately controlled by pressure changes induced in the combustion tube by the vaporisation and burning of the sample. The movement of the heater is arrested by a pressure switch when the pressure exceeds the equilibrium

pressure established by a choking plug, and its movement is resumed when the pressure drops again. Adequate contact time and uniform heating cycles are thus ensured. The apparatus can be used for solids and liquids and the principle is applicable to the Dumas micro-determination of N.

J. P. STERN

1048. **New method for the determination of oxygen isotopes in organic compounds.** H. Dahn, H. Moll and R. Menassé (Org.-Chem. Anst., Basel Univ.). *Helv. Chim. Acta*, 1959, **42** (4), 1225–1236.—Compounds containing ^{18}O (10 to 30 mg) are heated with *o*-phenylenediamine hydrochloride (50 to 100 mg) for 3 hr. at 300° ± 10°, to yield water, which is then equilibrated with a known amount of isotopically normal CO_2; the content of $CO^{18}O$ in the latter is then determined in a mass spectrometer. Compounds labelled in the carbonyl and alkoxyl groups can be differentiated by determining the total amount of ^{18}O by this method, and then determining the ^{18}O in the alkoxyl groups alone by treatment with "aniline magnesium bromide," which forms the anilides without affecting the carbonyl groups. The method is applicable to a large variety of compounds, but not to simple phenols and phenol ethers. An apparatus is described for the exact measurement of the CO_2 added to the reaction mixture. Results for 50 compounds are in good agreement with the calculated values.

J. L. PROSSER

1049. **Detection of nitrogen during the determination of carbon and hydrogen.** E. Abramson (Fac. de Méd., 45 rue des Saints-Pères, Paris). *Mikrochim. Acta*, 1959, (5), 770–771 (in French).—The test depends on the oxidation reaction of diphenylamine in H_2SO_4 soln. which, in the presence of NO_3^- and NO_2^-, forms diphenylbenzidine (**I**) and is finally converted into a blue quinonoid complex. *Procedure*—Between the two absorbents used in a carbon and hydrogen determination insert a small absorber containing alumina saturated with an H_2SO_4 soln. of **I**. The least trace of N is indicated by a blue ring at the point of entry of the gas. To avoid the retention of CO_2 it is necessary to use alumina of chromatographic quality.

D. F. PHILLIPS

1050. **The micro-determination of traces of nitrogen in organic substances by the modified Pregl - Dumas method.** E. Abramson and J. Laurent (Fac. de Méd., 45 rue des Saints-Pères, Paris). *Mikrochim. Acta*, 1959, (5), 786–791 (in French).—The use of a micro-nitrometer having a capacity of 0·25 ml and graduated in microlitres enables traces of N to be determined. A rapid technique is described for the determination of N in a 1 to 2-mg sample, which gives results within 0·2% of the theoretical.

D. F. PHILLIPS

1051. **Semi-micro method for the determination of fluorine and phosphorus in organic compounds containing both these elements.** C. Eger and J. Lipke (Sci. Dept., Min. of Defence, Tel-Aviv, Israel). *Anal. Chim. Acta*, 1959, **20** (6), 548–555.—The F is determined by a method already published (*cf.* Eger and Yarden, *Anal. Abstr.*, 1956, **3**, 2759), but since PO_4^{3-} in the melt from the Parr bomb interfere in the titration of F^- these ions are separated by ion exchange. The soln. (100 ml) of the melt is passed in turn through a column (250 mm × 27 mm) of Dowex 50-X4 cation-exchange resin (H^+ form) and a column (200 mm × 17 mm) of Amberlite IRA-400 anion-exchange resin (OH^- form). The F^- are removed from the Amberlite IRA-400 column by passing 600 ml of 0·25 *N* NaOH. This eluate is passed through a column (250 mm × 27 mm) of Dowex 50-X4; the first 100 ml of percolate is rejected, and the F^- are titrated in an aliquot of the remainder. The PO_4^{3-} are removed from the Amberlite IRA-400 column by passing 450 ml of *N* NaOH, the eluate is acidified with H_2SO_4 and the PO_4^{3-} are determined by the colorimetric method of Bernhart and Wreath (*cf. Anal. Abstr.*, 1955, **2**, 2078). Alternatively, the PO_4^{3-} can be determined by the same method in an aliquot of the original soln. without removal of F^-.

H. N. S.

1052. **Micro-determination of unsaturation in organic compounds.** M. R. Chaphekar and T. S. Gore (Univ. of Bombay, Matunga, India). *Mikrochim. Acta*, 1959, (5), 664–669 (in English).—A recently described apparatus (*cf.* Arjungi *et al.*, *J. Sci. Ind. Res., B, India*, 1958, **17**, 459), based on that of Roth and used by one of the authors for the micro-determination of active hydrogen in organic compounds, is now applied to the micro-determination of unsaturation by means of palladium on activated carbon catalyst in acetic acid. Hydrogen numbers in good agreement with computed ones were obtained and the precision was satisfactory.

D. F. PHILLIPS

1053. **Non-aqueous titration of phenolic hydroxyl.** E. J. Greenhow and J. W. Smith (C.S.I.R.O., Coal Res. Sect., P.O. Box No. 3, Chatswood, N.S.W., Australia). *Analyst*, 1959, **84**, 457–461.—Of three solvent - electrode systems, with 0·2 *N* Na aminoethoxide as titrant, a system of pyridine with an antimony indicating electrode and platinum reference electrode was selected, the procedure being similar to that of Katz and Glenn (*Anal. Chem.*, 1952, **24**, 1157) with the apparatus previously described (Brooks and Maher, *Fuel*, 1957, **36**, 51; Greenhow and Smith, *Ibid.*, 1958, **37**, 354). The sample (≃0·1 g), dissolved or suspended in 20 ml of solvent, was titrated potentiometrically in an atmosphere of N with 0·1-ml portions of 0·2 *N* Na aminoethoxide in ethylenediamine, the sample being stirred after each addition until the millivoltmeter reading was steady. When pptn. of sodium salts of weak phenols (*e.g.*, catechol and quinol) occurred, 2:6-xylenol was added. Tests on low-temp. tars showed that titrations of these would include dihydroxybenzenes with the phenol and cresols. Application of the method to the hydroxyquinones of naphthalene and anthracene was investigated. The apparent titration of simple quinones is ascribed to reaction with the titrant. If phenolic esters, lactones of phenols and certain quinones are absent, the procedure is suitable for the determination of mono- and poly-hydric phenolic hydroxyl in coal tar and pitch.

A. O. JONES

1054. **Determination of alkoxyl groups.** I. A. Favorskaya and É. M. Auvinen. *Vestn. Leningr. Univ.*, No. 16, *Ser. Fiz. i Khim.*, 1959, (3), 119–127.—A survey is given of modern methods for the determination of alkoxyl groups, with special reference to the determination of methoxyl, ethoxyl and *iso*propoxyl groups. (11 references.)

W. ROUBO

1055. **Quantitative separation and identification of alkoxyl groups by gas - liquid chromatography.** K. Kratzl and K. Gruber. *Monatsh. Chem.*, 1958,

89, 618–624.—The separation and identification of the alkyl iodides (**I**) obtained in the Zeisel procedure are carried out in 2 hr. by gas chromatography. The alkyl ether ($\simeq$10 mg) is decomposed in a modified Zeisel apparatus with CO_2 or N as the carrier gas and the **I** is absorbed in tri-*m*-tolyl phosphate (**II**) or polyoxyethylene glycol 400. **I** from C_1 to C_5 are easily separated from **II** and distinguished from each other at 84° with a gas flow of 20 ml per min. Chromatographic determinations of methoxyl, ethoxyl and propoxyl groups were made with an accuracy of $\pm$1% on 4-ethoxy-3-methoxybenzaldehyde and 3-methoxy-4-propoxybenzaldehyde. Chromatograms are also given for 3-(*o*-methoxyphenoxy)propane-1:2-diol, de-resinified spruce wood, hydroxyethyl-lignin and wood residue after ethanolysis. CHEM. ABSTR.

1056. **Micro-determination of methoxyl and ethoxyl.** R. F. Makens, R. L. Lothringer and R. A. Donia (Michigan Coll. of Mining and Technol., Houghton, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1265–1267.—Methoxyl and ethoxyl groups in samples containing both are converted into methyl and ethyl iodides and the iodides are separated by selective dissolution in trimethylamine - nitrobenzene. The separated iodides are determined by a modified Volhard or other suitable method. *Procedure*—Weigh 5 to 15 mg of sample into a tinfoil container and place into the alkoxyl apparatus. Partly fill the bubble counter with a suspension of red phosphorus in 5% $CdSO_4$ soln. and fill the two absorption tubes with 4 ml and 3 ml of 0·5 *N* trimethylamine in nitrobenzene, respectively. Add 3 to 4 drops of propionic anhydride (Elek, *Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 174) and phenol soln. to the reaction vessel, followed by 4 ml of HI (sp. gr. 1·7). Pass CO_2 (1 bubble per sec.), heat slowly to 140° and maintain for 2 hr. Cool under CO_2 for 1 hr. The tetramethylammonium iodide has then crystallised out, while ethyltrimethylammonium iodide remains in soln. in the absorption tubes. Two volumetric methods for determining the iodides are described.

J. P. STERN

1057. **Industrial analytical applications of gas chromatography. I. Analysis of impurities in methane.** M. De Vita, G. Caprioli and E. Pavan (Sicedison, Milan). *Chim. e Ind.*, 1959, **41** (4), 292–294.—A procedure is described for the determination of small amounts of CO_2, ethane, ethylene, propane, propene, *iso*butane, *n*-butane, and butenes in technical methane. The stationary phase is Celite C22, impregnated with 35% of a mixture of tri*iso*butylene and transformer oil. Retention times are listed. C. A. FINCH

1058. **Field method for the determination of phosgene.** B. E. Dixon and G. C. Hands (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1959, **84**, 463–464.—Strips of Whatman No. 1 filter-paper are soaked in a soln. containing 2% w/v of 4-*p*-nitrobenzylpyridine and 4% w/v of N-benzylaniline in benzene and dried in air. The paper is placed in a holder that exposes a circle of 1 cm diameter, and 120 ml of air is drawn through at $>$3 ml per sec. The red stain is compared with standard stains representing 0·25 to 10 p.p.m. of phosgene or with Lovibond glass colour-standards. If Cl is present it is removed by interposing on the inlet side of the paper another paper that has been soaked in an aq. soln. of 4% w/v of NaI and 10% w/v of $Na_2S_2O_3$. The presence of benzyl chloride, trichloroethylene or $CHCl_3$ (200 to 300 p.p.m.) has no effect on the formation of the stain. Benzoyl chloride produces a stain which, however, is transient in the presence of phosgene. Acetyl chloride reduces the intensity of the stain and, in concn. of 140 p.p.m., suppresses it. The test is sensitive to an atmosphere containing 0·25 p.p.m. of phosgene, and the stain is stable for at least 3 hr. A. O. JONES

1059. **Micro-detection of ethanol.** Shinichi Takagi. *Sci. and Crime Detection, Japan*, 1958, **11** (4), 22–25.—Potassium dichromate soln. (10%) (10 ml) and 5 *N* H_2SO_4 (5 ml) are mixed with the sample soln. in a 100-ml flask. A rubber bung with a glass tube containing a piece of cotton wool is put into the neck of the flask after the cotton wool has been moistened with 1 drop of 20% Na_2CO_3 soln., 1 drop of 1% phenylmethylaminopropane hydrochloride soln. and 2 drops of 1% sodium nitroprusside soln. The soln. is boiled for 5 min. and a blue colour is produced if ethanol is present. The limit of detection is 0·0015 ml of ethanol per 0·1 ml of sample soln. Esters, such as ethyl acetate and malathion, gave positive reactions. CHEM. ABSTR.

1060. **Paper electrophoresis of mono- and polyhydric alcohols.** H. Berbalk (Inst. Org. Chem., Tech. Hochsch., Wien). *Monatsh. Chem.*, 1959, **90** (1), 24–28.—The migration distances of the xanthates of 13 saturated aliphatic alcohols, 3 aromatic alcohols, furfuryl alcohol and 4 glycols have been compared with that of methanol (= 100); for normal primary alcohols these values decrease with increasing number of C atoms. Ethanediol and *n*-pentanethiol are readily separated as xanthates. N. E.

1061. **Determination of glycols by polarography.** Kiyoshi Takiura and Kyoko Koizumi (Pharm. Fac., Osaka Univ., Hotarugaike, Toyonaka). *J. Pharm. Soc. Japan*, 1958, **78** (9), 961–964.—The method of Souchay for the polarography of IO_3^- and IO_4^- (*Anal. Chim. Acta*, 1948, **2**, 17) is applied to the analysis of glycols. Sørensen's phosphate buffer (pH 7) is the best medium both for the polarography and for the oxidation with KIO_4 of straight-chain polyhydric alcohols, pentoses and hexoses. The sample soln. (10^{-3} *M*, 1 ml) is mixed with KIO_4 soln. (0·001 *M* in the buffer, 10 ml), set aside for 1 hr. at room temp., then mixed with gelatin soln. (1%, 0·1 ml) and the polarogram is recorded.

K. SAITO

1062. **Quantitative paper-chromatographic determination of sugars.** P. Albers and R. Freiskorn (Chemotherapeutic Res. Inst., "Georg-Speyer-Haus," Frankfurt a. M., Germany). *Annalen*, 1959, **622**, 150–159.—A Zeiss recording photometer is used to determine the extinction of the sugar spots (the areas of which are determined planimetrically) after treatment with aniline phthalate. The total "extinction area" is proportional to the square root of the concn. of the sugar ($>$150 μg of arabinose, mannose, galactose or glucose, or of mixtures thereof) when opaque paper and an FE54 filter are used. Calibration curves are drawn, and the mean of 12 to 20 determinations gives the sugar concn. with an accuracy to within $\pm$1%. *Procedure*—Separate the sugars (0·02 ml of soln. containing 25 to 150 μg) by ascending chromatography on Whatman No. 4 paper, with *n*-butanol - pyridine - water (3:1:1) or phenol - water (4:1 or 17:3) as solvent. Spray the dry

paper with a soln. containing 1·86 g of aniline, 3·38 g of phthalic acid, 50 ml of *n*-butanol, 40 ml of acetone and 10 ml of H_2O. Dry at 85° for 40 min. (hexoses give brown, pentoses reddish, spots). Cut into 4-cm strips and measure the extinction and the area of the spot; read the amount of sugar present from the appropriate calibration curve. For single substances take the mean of 12 values, for mixtures, that of 20 values. J. P. STERN

1063. **Sensitive colorimetric method for determination of fructose.** L. E. Hessler (Texas Technol. Coll., Lubbock, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1234–1236.—Fructose (0·05 to 0·3 mg) (**I**) is determined colorimetrically at 450 mμ in the presence of aldose sugars after treatment with *p*-anisidine (**II**) or *o*-dianisidine (**III**) in 85% H_3PO_4. The method is used for **I** in cotton boll, and 0·1 mg of **I** is determined with standard deviations of 0·00258 and 0·00152 mg and coeff. of variation of 2·7% and 1·5%, with **II** and **III**, respectively. *Procedure*—Dilute the sample (containing 0·05 to 0·3 mg of **I**) with H_2O to 2 ml, add 4 ml of a 0·5% soln. of **II** or **III** in 85% H_3PO_4; boil the soln., then cool it to room temp., and measure the extinction at 450 mμ after 30 min. J. P. STERN

1064. **Method for determinating fructose and glucose in the presence of each other.** E. Trifirò and D. Proto. *Ind. Conserve, Parma*, 1959, **34** (3), 201–208.—*Procedure*—The soln. containing from 0·25 to 0·75 mg of reducing sugars is placed in a 100-ml flask with 5 ml of alkaline $K_3Fe(CN)_6$ soln. [150 g of $NaH_2PO_4.12H_2O$, 160 g of Na_2CO_3 and 4 g of $K_3Fe(CN)_6$ made up to 1 litre]. The flask is heated on a boiling-water bath for 30 min., then cooled, and 10 ml of 2 *N* H_2SO_4 and 4 ml of molybdo-arsenic acid reagent are added. The blue colour developed is read at 750 mμ; the total reducing sugars are thus obtained. On another sample a reduction is carried out at 60° for 45 min.; only the apparent glucose is then determined. A control containing glucose and fructose in known concn. must be carried through the same procedure to determine the amount of unoxidised glucose, and the true concn. of glucose is then calculated.

L. ZANONI

1065. **Electrophoresis. IV. Paper electrophoresis of aliphatic aldehydes.** H. Berbalk. *Monatsh. Chem.*, 1958, **89**, 536–539.—The method is based on the conversion of aldehydes into hydroxamic acids; it is rapid, and ketones do not interfere. The aldehydes in water or aq. methanol are warmed with a granule of N-hydroxybenzenesulphonamide (**I**) and 2 *N* NaOH (1 ml) to give a soln. of the sodium salts of the hydroxamic acids, and this is transferred to filter-paper strips satd. with 0·1 *N* NaOH and subjected to paper electrophoresis. The separated hydroxamic acids appear as violet spots on a white background when sprayed with a soln. of 1·5% $FeCl_3$ in dil. HCl. The by-product benzenesulphinic acid gives yellow spots with $FeCl_3$ which migrate so much faster than the hydroxamic acids that they cause no interference. Aromatic aldehydes that react with **I** can also be determined in this way. The migration velocity of formaldehyde is 12×10^{-5} sq. cm per sec. per V. The transport range relative to formaldehyde, W_f, on S. & S. 2043*b* filter-paper with 0·1 *N* NaOH as a transport medium is given—formaldehyde 100, propionaldehyde 64, *n*-butyraldehyde 57, *iso*valeraldehyde 59, *n*-hexaldehyde 52, 2-ethylbutyraldehyde 44, *n*-heptaldehyde 47, octaldehyde 41, 2-ethylhexaldehyde 47, *n*-decaldehyde 30, lauraldehyde 13, crotonaldehyde 66, citral 46, citronellal 81, benzaldehyde 67 and cinnamaldehyde 44. CHEM. ABSTR.

1066. **Rapid colorimetric method for determining glyoxal.** C. S. Wise, C. L. Mehltretter and J. W. Van Cleve (N. Utilization Res. and Devel. Div., U.S. Dept. of Agric., Peoria, Ill.). *Anal. Chem.*, 1959, **31** (7), 1241–1242.—The glyoxal formed during the hydrolysis of starch oxidised with IO_4^- is determined colorimetrically at 550 mμ after reaction with 1:2-dianilinoethane dihydrochloride (**I**) with an accuracy within ±1·5%. Formaldehyde, acetaldehyde, glucose and erythrose do not interfere. *Procedure*—Treat a 3-ml aliquot of sample (containing ≃1·2 mg of glyoxal) with a 1·33% soln. of **I** in 95% ethanol (3 ml) and 0·6 ml of conc. HCl. Heat at 77° for 30 min., dilute to 100 ml with 95% ethanol, and measure the extinction of a 15-ml aliquot at 550 mμ against a blank. A reference standard should be run simultaneously.

J. P. STERN

1067. **Polarographic determination of acetone.** R. E. Van Atta and D. R. Jamieson (Dept. of Chem., Univ. of S. Illinois, Carbondale, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1217–1219.—The product of the reaction between acetone (**I**) and *n*-butylamine (**II**) is suitable for the rapid routine determination of **I** in the concn. range from 0·05 to 100%. The equilibrium constant for the reaction between **I** and **II** is $8·4 \times 10^{-2}$. The accuracy is within ±1%. The method is used for determining **I** in the presence of benzophenone, benzhydrol, *iso*propyl alcohol and Al *iso*propoxide. *Procedure*—To the soln. of **I** (1 ml), diluted if necessary, add 5 *M* NH_4Cl (20 ml) and pure **II** (17·0 ml), and dilute to 100 ml. Measure the current at −1·45 and −1·75 *V vs.* the S.C.E., determine the difference, and obtain the concn. of **I** from a calibration curve.

J. P. STERN

1068. **Comparison of transmittance and reflection spectra of the 2:4-dinitrophenylhydrazones of acetone and 4-methylpentan-2-one.** H. Zeitlin and A. Niimoto (Chem. Dept., Univ. of Hawaii, Honolulu). *Anal. Chem.*, 1959, **31** (7), 1167–1170.—The reflection spectra of these compounds in the powdered state have been compared with the transmittance spectra in ethanol. Similar comparisons have been made with these compounds adsorbed as solids and in soln. on various types and grades of Al_2O_3, silica gel and filter-paper. Spectral characteristics are retained, in most cases, in reflectance, although relatively constant bathochromic displacements take place in passing from transmittance to reflectance. These displacements are due to the state of subdivision of the compounds.

K. A. PROCTOR

1069. **Determination and removal of small traces of water in acetone.** K. Jordan and W. R. Fischer (Versuchsanstalt d. Fried. Krupp WIDA-Fabrik, Essen, Germany). *Z. anal. Chem.*, 1959, **168** (3), 182–190.—Small amounts (0·125 to 0·25% by vol.) of H_2O in acetone may be determined chemically by an adaptation of the acetyl chloride method (Smith and Bryant, *J. Amer. Chem. Soc.*, 1935, **57**, 841) or, preferably, physically by measuring the temp. at which a mixture of the sample (20·0 ml) and light petroleum (20·0 ml) becomes homogeneous. The solubility curve is rectilinear for

H_2O contents $<1{\cdot}75\%$, and 0·1° corresponds to a variation of 0·01% of H_2O (absolute). The accuracy of both methods is within $\pm 0{\cdot}01\%$ (absolute). Acetone kept over anhyd. K_2CO_3 or $CaCl_2$ contains 0·2% of H_2O. Treatment with acetyl chloride in pyridine affords acetone containing $<0{\cdot}005\%$ by vol. of H_2O, which is highly hygroscopic.

J. P. STERN

1070. **Analysis of nitroparaffins by the use of a chromatographic method.** M. I. Rozova and F. N. Stolyarova (State Inst. of Appl. Chem., Leningrad). *Zhur. Anal. Khim.*, 1959, **14** (3), 343–346.—Nitroparaffins (C_1 to C_3) are reduced to the corresponding amines and chromatographed on a column of starch (35 g) and anhydrous CaO (0·35 g), hydrated to contain 60% of H_2O. Benzene (28 ml) is added and a column (18 cm × 17 to 18 mm) is prepared. A layer (1·5 to 2 cm long) of starch (2 g), hydrated CaO (0·9 g) and H_2O (1 ml) is placed above the column. An aq. soln. of the amine (1 ml) is added and the chromatogram is developed with butanol - benzene (3:7). The emerging fractions are titrated in aq. soln. with 0·05 *N* HCl to bromothymol blue.

K. R. C.

1071. **Titration of ketimines in glacial acetic acid.** P. L. Pickard and F. A. Iddings (Univ. of Oklahoma, Norman, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1228–1230.—Ketimines in amounts $>0{\cdot}06$ milli-equiv. are determined rapidly with an accuracy within $\pm 1\%$ by titration with 0·1 *N* $HClO_4$ in glacial acetic acid, either potentiometrically or with crystal violet as indicator. Alkyl and aryl halides do not interfere, but NH_3, amines and $>1\%$ of nitrile cause erratic results.

J. P. STERN

1072. **Polarographic determination of nitrilotriacetic acid in (ethylenedinitrilo)tetra-acetic acid [EDTA].** R. L. Daniel and R. B. LeBlanc (Dow Chemical Co., Freeport, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1221–1223.—Of the metal chelates of nitrilotriacetic acid (**I**) and EDTA (**II**) those of Cd^{II} and Ni^{II} differ most in stability and the complexes of Cd^{II} are suitable for the polarographic determination at pH 10 of 0·03% by wt. of **I** as a contaminant of **II**. The accuracy is within $\pm 5\%$ of the amount present or $\pm 0{\cdot}03\%$ (absolute), whichever is greater. *Procedure*—Dissolve the sample (5 g for 0·1 to 3% of **I**) in H_2O (75 ml) and adjust to pH 10 with *M* HNO_3 or *M* KOH. Add *M* $Cd(NO_3)_2$ dropwise until the pH changes by $<0{\cdot}1$ per drop, and then add two more drops. Re-adjust the pH to 10 with *M* KOH and dilute to 100 ml. Dilute 10 ml of this soln. to 100 ml and polarograph from −0·4 to −1·3 V. Measure the height of the Cd - **I** wave ($E_{\frac{1}{2}}$ = −0·91 V) and compare it with a calibration curve.

J. P. STERN

1073. **Nephelometric determination of xanthates in solution.** I. N. Plaksin, N. A. Suvorovskaya and M. M. Voskresenskaya. *Sb. Nauch. Trud. Mosk. Inst. Tsvet. Met. i Zolota*, 1958, (31), 305–307; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,625.—The nephelometric determination of butylxanthate as the coloured compound with Ni $[(C_4H_9O{\cdot}CS{\cdot}S)_2Ni]$ is suggested and studied. It is shown that the use of standard soln. of concn. lower than that of the test soln. leads to high results; excess of Ni does not influence the accuracy of the determination. To stabilise the soln. it is recommended that starch be added as protective colloid. Cupric ions and Zn^{2+} interfere; Pb does not.

C. D. KOPKIN

1074. **Detection and estimation of thiosulphinates and thiosulphonates.** D. Barnard and E. R. Cole (British Rubber Producers' Res. Assoc., Welwyn Garden City, Herts., England). *Anal. Chim. Acta*, 1959, **20** (6), 540–547.—The paper-chromatographic method of Lynn *et al.* (*Anal. Chem.*, 1956, **28**, 132) is applied to the separation of alkyl- and aryl-thiosulphinates from similarly substituted thiosulphonates and to the separation of different members of each series. Methods for locating the spots are described. Thiosulphinates are determined by titrating the iodine they liberate from KI in acetic acid soln. Thiosulphonates interfere, but the interference is suppressed by a large preponderance of thiosulphinate. Thiosulphonates are determined by mixing the sample with an excess of thiophenol in ethanol and titrating the resulting sulphinic acid with 0·1 *N* or 0·02 *N* NaOH. Thiosulphinates do not interfere.

H. N. S.

1075. **Spectroscopic techniques for identification of organosilicon compounds.** A. L. Smith and J. A. McHard (Dow Corning Corp., Midland, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1174–1179.—The use of i.r. spectroscopic techniques for the identification and characterisation of organosilicon monomers and polymers is discussed. Possible applications of u.v., mass, Raman, emission and nuclear magnetic resonance spectroscopy are also reviewed. (58 references.)

K. A. PROCTOR

1076. **Raman spectra of hexachloro*cyclo*hexane isomers and the quantitative determination of the γ-isomer.** P. Chiorboli and C. Gualandi. *Boll. Sci. Fac. Chim. Bologna*, 1959, **17** (1), 9–14.—The Raman spectra of the hexachloro*cyclo*hexane isomers (α, β, γ, δ) in soln. in various solvents (α- in dioxan, β- in acetone, γ- in benzene and in dioxan, δ- in dioxan) have been obtained by mean of photoelectric and photographic recording. The determination of the γ-isomer in a mixture of other isomers has been carried out after the construction of a calibration curve with a benzene soln. of the pure γ-isomer. The analytical line used is 343 cm^{-1}, the intensity of which is compared with that of the CCl_4 line at 459 cm^{-1} by the method of external comparison. The errors were $<3{\cdot}5\%$.

L. ZANONI

1077. **Polarographic determination of 1:2-diamino*cyclo*hexane in hexamethylenediamine.** M. E. Hall (Chemstrand Corp., Decatur, Ala., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1219–1221.—Nickel and Co form reducible complexes with 1:2-diamino*cyclo*hexane (**I**), but not with hexamethylenediamine (**II**) at high pH; Cu^{II} forms complexes with both. The nickel complex at high concn. of **I** (40 mg per 100 ml) gives two reduction waves and at low concn. (≯4 mg per 100 ml) one reduction wave, and is suitable for the determination of **I** as a contaminant of **II**. Substances that form similar nickel complexes at pH $>11{\cdot}7$ may interfere when present in high concn., but NH_4^+ do not interfere. The coeff. of variation is $\pm 5\%$ on 0·3 to 1 mg of **I** in 25 ml of electrolyte, and $\pm 10\%$ for lower concn. *Procedure*—To the sample (≯4 g) add water (10 ml) and 2% $NiCl_2$ soln. (1 ml) and dilute to 25 ml. Mix, and centrifuge to remove any pptd. $Ni(OH)_2$; transfer the clear liquid to an H-type cell with a S.C.E. in one limb, remove O, and record the polarogram between −0·7 and −1·3 V at 25°. Measure the diffusion current and determine the content of **I** from a calibration curve.

J. P. STERN

1078. **Organic chemical microscopy. V. The photomicrography and crystallography of aromatic halide derivatives.** R. E. Dunbar and W. M. King (N. Dakota State Coll., Fargo, U.S.A.). *Microchem. J.*, 1959, **3** (2), 143–153.—Thirty aromatic halides were investigated with regard to the preparation of derivatives suitable for crystallographic identification. A total of nine aryl halides and seven halophenols were converted into their mono- and dinitro derivatives. The twelve monohalotoluenes were oxidised to the corresponding benzoic acids. Two monochloro- and dichloro-xylenes were oxidised to the corresponding monochloro- and dichloroterephthalic acids. Photomicrographs are given for each aromatic halide investigated. The use of a petrological microscope in the identification of organic derivatives is recommended as an easy but important addition to the normal m.p. procedure.
D. F. PHILLIPS

1079. **Isomer ratio analyser for toluene*di*isocyanate [dicyanatotoluene] based on dielectric-constant measurements.** S. Steingiser, W. C. Darr and E. E. Hardy (Res. Dept., Mobay Chem. Co., New Martinsville, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1261–1265.—The method described is applicable only to pure systems consisting of 2:4-dicyanatotoluene and 2:6-dicyanatotoluene. The analyser can be used continuously and the accuracy is within $\simeq \pm 0{\cdot}2\%$ of an isomer unit, better than methods previously used. The effects of solvents, temp., and certain interfering substances are reported.
K. A. PROCTOR

1080. **Polarographic determination of small amounts of phenol with a rotating platinum anode.** V. I. Ginzburg and T. A. Frishman (Sci.-Res. Inst. of Synthetic Alcohols and Org. Products, Moscow). *Zhur. Anal. Khim.*, 1959, **14** (3), 336–342.—The optimum conditions are described for the preparation of a rotating platinum electrode for the production of clear, reproducible phenol polarograms corresponding to large limiting currents; small amounts of phenol are determined with a supporting electrolyte of 0·1 *M* $NaNO_3$ - 0·1 *M* H_2BO_3 - NaOH at pH 10·7 ($\pm$0·1) and an aq. soln. of $1{\cdot}0 \times 10^{-3}$ *M* phenol serves as the standard soln. The method can be used for the direct determination of up to $1{\cdot}0 \times 10^{-5}$ *M* phenol in coloured turbid media containing other organic impurities, *e.g.*, α-methylstyrene, acetophenone and acetone. The coeff. of variation is 2·5 to 4·7%.
K. R. C.

1081. **Quantitative analysis of commercial bisphenol A by paper chromatography.** W. M. Anderson, G. B. Carter and A. J. Landua (Res. Lab., Shell Chem. Corp., Houston, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1214–1217.—The three impurities in commercial bisphenol A [2:2-di-(*p*-hydroxyphenyl)propane] (**I**) and the content of **I** are determined by preparing two linear paper-chromatograms (one irrigated by H_2O, the other by CCl_4), spraying with a coupling agent (*p*-nitrobenzenediazonium fluoroborate in acetone), and measuring the areas of the coloured spots with a planimeter. Standard mixtures are run simultaneously. The sample composition affects the R_F values. For the CCl_4 chromatograms, equilibration with H_2O vapour is desirable. On 100 μg of sample the reproducibility is generally within $\pm$1% for all four components.
J. P. STERN

1082. **Application of proton resonance to the elucidation of structure and to the identification of natural and synthetic quinones with isoprene side-chains.** C. von Planta, E. Billeter and M. Kofler (F. Hoffmann-La Roche & Co. A.-G., Basle). *Helv. Chim. Acta*, 1959, **42** (4), 1278–1282.—Proton-resonance spectra are reported for vitamins K_1 and K_2, ubiquinone and quinone, and the contribution of the various structural elements is discussed.
J. L. PROSSER

1083. **Infra-red absorption method for the determination of *cyclo*hexanone and *cyclo*hexanol in *cyclo*hexane oxidation products.** I. V. Berezin, N. F. Kazanskaya and G. B. Meluzova (M. V. Lomonosov State Univ., Moscow, USSR). *Zhur. Fiz. Khim.*, 1958, **32**, 1218–1224.—*cyclo*Hexanone and *cyclo*hexanol are the principal oxidation products of *cyclo*hexane, and an i.r. absorption method for their determination to within $\pm$3% was developed by using a 0·5-ml sample; the time taken is $<$10 min. The cell used was of a special design, and an automatic slit-width regulator and recorder of the intensity of the absorption bands enabled determinations of several components to be made with a single cell. Several *cyclo*hexanone and *cyclo*hexanol absorption-band widths were determined in *cyclo*hexane soln. and the effect of the slit width on the values was studied.
CHEM. ABSTR.

1084. **Additive effect of substituent groupings on the chromatographic behaviour of phenoxyacetic acids.** L. S. Bark and R. J. T. Graham (Chem. Dept., Derby and District Coll. of Technol., Green Lane, Derby, England). *Analyst*, 1959, **84**, 454–455.—The behaviour of phenoxyacetic and some substituted phenoxyacetic acids in various solvent mixtures containing *n*-butanol was investigated. A 0·0025-ml portion of a 1% soln. of each was submitted to ascending paper chromatography on paper previously treated with NH_3 vapour. The chromatograms were developed with a 0·04% soln. of bromocresol purple in a mixture of formaldehyde and abs. ethanol (1:5), and were then exposed to NH_3 vapour. The acids appeared as yellow spots on a purple ground. The definition and shape of the spots varied with different solvents. Graphs of R_M, *i.e.*, $\log_{10}[(1 - R_F)/R_F]$, against the number of substituent groups were linear and, for the series investigated, R_F values increased with the amount of NH_3 in the solvent system. The three solvents used were *n*-butanol (*a*) saturated with water; (*b*) saturated with 1·5 *N* aq. NH_3; and (*c*) saturated with 3 *N* aq. NH_3. Results are tabulated.
A. O. JONES

1085. **Detection of primary aromatic amines.** E. Sawicki, T. W. Stanley and T. R. Hauser (R. A. Taft Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati, Ohio). *Chemist Analyst*, 1959, **48** (2), 30–31.—A colour test with azobenzene-4-diazonium fluoroborate (**I**) (prep. described) carried out in dimethylformamide (**II**) or dimethyl sulphoxide (**III**) is applied to the detection and determination of aromatic primary amines, particularly those that are harmful air pollutants, *e.g.*, 2-naphthylamine, benzidine and 2-aminofluorene. *Spectral test procedure*—A 0·5% soln. (0·1 ml) of **I** in either **II** or **III** containing a small amount of conc. HCl is added to a soln. of the sample (10 to 100 μg of aromatic amine) in the same solvent. After 5 min. 0·2 ml of 10% aq. tetraethylammonium hydroxide soln. (**IV**) is added and the mixture is diluted to 10 ml. The absorption spectrum in the region 400 to 800 mμ is then determined. *Spot*

test procedure—Higher sensitivity is attained by using **III** as solvent for both sample and reagent. One drop of 0·25% reagent soln. is added to 1 drop of sample soln. on a porcelain spot-plate. After 3 to 5 min., 1 drop of **IV** is added with stirring. A positive reaction is usually indicated by a violet colour. Results with 9 amines are tabulated for both spectral and spot tests. Phenols and aromatic secondary amines interfere. Phenols can be removed by alkaline extraction and the presence of secondary amines may be confirmed by the piperonylidene chloride test, but interference by secondary amines is only slight. S. M. MARSH

1086. **Determination of small amounts of α-picoline in pyridine by a solution-temperature method.** K. A. Adey and J. D. Cox (Midland Tar Distillers Ltd., Four Ashes, nr. Wolverhampton, England). *Analyst*, 1959, **84**, 414–417.—The pyridine sample (5 ± 0·01 g) and a KCl soln. (15 ± 0·05 g of a 20% w/v aq. soln.) are weighed into the inner tube of the standard apparatus ("Standard Methods for Testing Tar and its Products," 4th Ed., Gomersal, Leeds, 1957, p. 12) and the exact wt. of each component is recorded. A T5C standard thermometer (*op. cit.*, p. 321) is fitted and the stirred liquid is brought to 25°. The water bath is maintained throughout the determination at 27·8° ± 0·2°. The inner tube is inserted in the air jacket already in the bath and the liquid is stirred. The temp. at which the liquid develops a milky white opacity not dispersed by stirring is the solution temp., and this can be read to within 0·02°. The determination is repeated without disturbing the apparatus. The temp. is corrected by adding or subtracting 0·015° for each 0·01% w/w by which the sample concn. in the mixture exceeds or is less than 25%, respectively, and by subtracting 0·0085° for each 0·01% of water in the pyridine sample (determined by the Karl Fischer method). The amount of α-picoline present is $(27{\cdot}0 - t)/0{\cdot}7\%$ w/w, where t is the solution temp.

A. O. JONES

1087. **Spectrophotometric determination of the nitrile of isonicotinic acid by the colour reaction with sodium nitroprusside.** W. Czerwiński (Inst. of Gen. Chem., Warsaw, Poland). *Chem. Anal., Warsaw*, 1958, **3** (1), 47–51.—The red colour produced with sodium nitroprusside and NaOH is measured at 480 mμ. Beer's law is obeyed up to 10 μg per ml. Nicotinonitrile gives a slight colour, and must be allowed for. The nitrile is separated from interfering substances (*e.g.*, *iso*-nicotinamide, picolines and NH_3) by steam-distillation from a neutral soln. H. DMOWSKA

1088. **Polarography of 2-mercaptobenzothiazole.** M. M. Bruno and R. L. Stoddard (Goodyear Atomic Corp., Portsmouth, Ohio). *U.S. Atomic Energy Comm.*, Rep. GAT-L-410, 1958, 7 pp.—A method for determining low concn. of 2-mercaptobenzothiazole in water is described. Dibenzothiazolyl disulphide interferes and is removed by pptn.

NUCL. SCI. ABSTR.

1089. **Determination of liquid hydrocarbons by gas chromatography.** D. A. Vyakhirev, M. I. Ostasheva and L. E. Reshetnikova. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 334–338; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,628.—Adsorption chromatography and partition gas-liquid chromatography are used to separate the components of "light oil" (the liquid product of the pyrolysis of kerosine) for the quant. determination of benzene (**I**), toluene (**II**) and xylene (**III**). Mixtures of **I**, **II**, **III**, *cyclo*hexane and heptane have been separated by adsorption chromatography on silica gel MSM (length of sorbent layer 150 cm, diam. 1·2 cm, rate of flow of N ≃ 150 ml per min.) with a chromathermograph with a moving furnace; the desorption temperatures are 88°, 120°, 150°, 64° and 79°, respectively. Separation of synthetic mixtures and light oil by partition gas-liquid chromatography was carried out on kieselguhr satd. with various high-boiling liquids, of which triethylene glycol was the most suitable. The conditions used were—length of sorbent layer 130 cm, diam. 0·5 cm, temp. 100° ± 1°, rate of flow of N 100 ml per min.

C. D. KOPKIN

1090. **Chromatographic resolution of petroleum porphyrin aggregates.** L. R. Fisher and H. N. Dunning (Bureau of Mines, U.S. Dept. of the Interior, Bartlesville, Okla.). *Anal. Chem.*, 1959, **31** (7), 1194–1197.—Polar and non-polar porphyrins can be separated rapidly by chromatography with partly deactivated alumina. The fractions obtained are characterised by paper chromatography and spectral analysis. K. A. PROCTOR

1091. **Direct determination of oxygen and its application in fuel analysis.** W. Radmacher and A. Hoverath (Brennstoffchem. Inst. der Ruhrkohlen-Beratung G.m.b.H., Essen, Germany). *BrennstChemie*, 1959, **40** (4), 97–104.—The method consists in carbonising the fuel, demineralised (except for pyrites) by the method of Radmacher and Mohrhauer (*cf. Anal. Abstr.*, 1957, **4**, 4036), followed by treatment with HF and HCl, and reducing the oxygenated products by contact with lampblack at 1120°. Special quartz tubes are described for drying the sample in a combustion boat at 125° and for carbonising it at 1050° in N without access of air. This temp. ensures the complete expulsion of O from the residue without causing reduction of residual oxides or quartz. The outlet gases pass through a train consisting of tubes containing copper wire at 800° (to ensure reduction of CO_2), NaOH, $Mg(ClO_4)_2$, I_2O_5 heated at 118° by a jacket of acetic acid, wadding impregnated with KI to absorb iodine, and a weighed soda-lime tube. The O, evolved from the sample as CO, is determined either gravimetrically as CO_2 or by titration of the liberated iodine with 0·02 *N* $Na_2S_2O_3$. Results by both methods and by different operators showed deviations from the mean from ±0·08 for 2·5% of O to ±0·2 for 9·2% of O. Silicate-bound H_2O in the mineral matter of the sample can be determined from results obtained on the sample before and after treatment with HF. The use of the method in examining the properties of coal is discussed.

A. R. PEARSON

1092. **Ranges of application of processes for the determination of hydrogen sulphide in coke oven gas.** P. Michaelis (Steinkohlenbergwerk Heinrich Robert A.-G., Hamm, Germany). *BrennstChemie*, 1959, **40** (4), 104–112.—The German Coal Mining Association has approved three methods for determining H_2S in coke oven gas at various stages of purification—(*a*) the Pb acetate paper test, (*b*) absorption in a soln. of Cd acetate in aq. acetic acid, and (*c*) absorption in aq. NaOH soln. Method (*a*) can be used for tests of 1 min. duration at H_2S concn. ⩾1·0 g per 100 cu. metre, within which

range the concn. can be judged from the depth of colour developed. The process can be adapted for recording by means of balanced photocells. Method (*b*) is used for long-period tests at all but very high H_2S concn. Rapid tests can also be carried out. Except at low concn., the pptd. CdS must be filtered off and washed before titration with 0·1 *N* iodine. The precision is within ±2% of the mean. By using $CdCl_2$ in aq. HCl the method can be adapted for recording by means of the change in conductivity due to the HCl liberated. Method (*c*) is used for long-period tests on foul gas and cannot be adapted for recording. The S^{2-} are oxidised to SO_4^{2-} by H_2O_2, and the soln. is titrated before and after passage through an ion-exchange column, in which Na^+ are replaced by H^+. Organic sulphur compounds do not interfere, and the products of side reactions (*e.g.*, SCN^-, $S_2O_3^{2-}$ and S_n^{2-}) are included in the oxidation, but NH_4Cl in the gas causes a positive error. Alternatively, an excess of 0·1 *N* iodine in aq. HCl may be added to an aliquot of the soln. and the excess of iodine back-titrated. Errors may arise from oxidative side reactions unless the titration is carried out promptly. If the concn. of NaOH is >10%, some yellowing due to the fixation of CS_2 as Na_2CS_3 may occur, and unsaturated hydrocarbons may be absorbed; these errors are minimised by adding an excess of conc. HCl before titrating, and, in short-period tests, by using dilute (0·5%) aq. NaOH soln. A. R. PEARSON

1093. **Qualitative analysis of organic pigments.** P. Rubbi (Lab. Ric. Silo, Turin). *Pitture e Vern.*, 1959, **15** (1), 65–68.—The pigment is boiled successively for a few minutes with (*i*) benzene, to dissolve neutral azo colouring matters, (*ii*) 2% Na_2CO_3 soln., to dissolve acid colouring matters, (*iii*) 10% HCl, to dissolve basic colouring matters and (*iv*) $Na_2S_2O_4$ soln. to dissolve anthraquinone pigments. The phthalocyanines are left as an insoluble residue. The identification of individual azo pigments is discussed, and tables are given showing the m.p., colours given with acid and alkali, and the coupling components of some of the commoner pigments of this class. The pigments may be decomposed to their coupling components by reduction with $SnCl_2$ or $Na_2S_2O_4$. D. R. DUNCAN

1094. **TAPPI routine control method RC271: Iron in cellulosic materials.** *TAPPI*, 1959, **42** (4), 147A.—For the determination of minute traces of Fe, the sample (10 g) is ashed in a silica or platinum dish, the residue is dissolved in HCl (1 : 1) (10 ml) and the soln. is transferred to a Nessler cylinder after reduction of the volume to 4 to 5 ml. The Fe is oxidised with 10% $(NH_4)_2S_2O_8$ soln. (sufficient to make the soln. ≃0·02 *M* in this reagent) and 10% KSCN soln. (5 ml) is added. After dilution of the soln. to 50 ml the colour is compared visually with a blank. R. AICHER

1095. **Quantitative determination of the sodium salt of carboxymethylcellulose.** M. Z. Finkel'shtein, I. M. Timokhin and Kh. U. Mukhamedov. *Izv. Vyssh. Uchebn. Zavedenil. Neft' i Gaz*, 1958, (12), 45–50; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,640.—Gravimetric and volumetric methods are proposed for determining the content of carboxymethylcellulose (**I**) in mortars for drilling. (*i*)—To an aq. soln. of **I** at pH 5 add a 1% soln. of $Al(NO_3)_3$ to precipitate **I** completely as the aluminium salt, warm to between 50° and 55°, collect the ppt. in a sintered glass filter, wash it, and dry it to const. wt. at 105°. The method gives low results (deviations from −1·82 to −5·67%). The addition of ethanol (20% of the vol. of the test soln.) improves the results. (*ii*)—Dissolve a sample of **I** (0·2 to 0·6 g) in 100 ml of water, add 20 ml of ethanol, *N* H_2SO_4 to pH 5·1, and 45 ml of 0·1 *N* $CuSO_4$. Adjust the pH to 3·35 and warm the soln. to between 50° and 55°. Filter off the ppt. of the copper salt of **I**, and wash it with 30% ethanol (2 × 20 ml); evaporate the ethanol from the filtrate, add 5 ml of 6 *N* acetic acid and 5 g of KI and titrate the liberated iodine with 0·1 *N* $Na_2S_2O_3$. The percentage of **I** (*a*) is given by the formula $a = (0{\cdot}016 + 3{\cdot}24/\gamma)(v_1 - v_2)100/b$, where *b* is the wt. of sample in g; v_1 and v_2 are the vol. of 0·1 *N* $CuSO_4$ and $Na_2S_2O_3$ in ml; γ is the degree of etherification of the **I**. The error is from +0·14 to −3·97%. The method is used for studying the adsorption of **I** by clay particles and is suitable for the determination of γ. C. D. KOPKIN

1096. **Modified 4-aminoantipyrine [4-aminophenazone] colorimetric method for phenols. Application to an acrylic monomer.** R. J. Lacoste, S. H. Venable and J. C. Stone (Rohm & Haas Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1246–1249.—The use of a borate buffer in place of aq. NH_3 - NH_4Cl enables phenols to be determined with 4-aminophenazone under conditions of widely varied ionic concn. Phenol (0·0003 to 1%) and *p*-methoxyphenol (0·0004 to 1%) can thus be determined colorimetrically in acrylic monomers. Quinol, NN′-diphenyl-*p*-phenylenediamine and *p*-hydroxydiphenylamine (*p*-anilinophenol) do not interfere. *Procedure*—Shake a 5-ml aliquot of monomer for 1 min. with 5·0 ml of 1·0 *N* NaOH. To 2·0 ml of the clear aq. layer add 18 ml of a soln. 0·4 *M* in H_3BO_3 and 0·1 *M* in NaOH, 0·1 ml of 3% aq. 4-aminophenazone soln. and 0·2 ml of 10% aq. $K_3Fe(CN)_6$ soln. Check the pH (9·1 to 9·2). Measure the extinction. J. P. STERN

1097. **Indirect polarographic analysis.** A. V. Ryabov and O. G. Tarakanov. *Trudy Khim. i Khim. Tekhnol.*, 1958, (2), 423–425; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,622.—A procedure is developed for the quant. polarographic determination of a mixture of methacrylic acid (**I**) and methyl methacrylate (**II**) in 0·1 *N* LiCl in 50% ethanol in the presence of gelatin. **I** gives a characteristic polarographic wave, but **II** is not reduced. Brominate the mixture of **I** and **II** with 3 *M* Br in methanol and destroy the excess of Br with aq. NH_3 while cooling. The bromo derivatives of **I** and **II** give combined polarographic curves with $E_{\frac{1}{2}}$ close to zero. In one sample of the mixture determine **I**, brominate a second sample, polarograph **I** and **II** together, then determine **II** by difference. C. D. KOPKIN

1098. **Polarographic determination of butyl methacrylate in plasticised poly(butyl methacrylate).** V. N. Dmitrieva and V. D. Bezuglyĭ (Kharkov Dental Materials Works). *Zavod. Lab.*, 1959, **25** (5), 555.—With a basal soln. of 0·02 *N* tetramethylammonium iodide in 94% methanol containing 4 to 6% of benzene, dibutyl phthalate gives two waves ($E_{\frac{1}{2}} = -1{\cdot}77$ and $-2{\cdot}06$ V) and butyl methacrylate gives one wave ($E_{\frac{1}{2}} = -1{\cdot}99$), which is superimposed on the second of the two waves of dibutyl phthalate. From the polarogram the contents of each ester can be determined. The sample (1 to 1·5 g) of plasticised polymer is dis-

solved during 3 to 4 hr. in 10 ml of benzene, the soln. is diluted with methanol, and an aliquot is taken. G. S. SMITH

1099. **Oligomers. XVII. Quantitative paper-chromatographic determination of secondary amines and amides.** H. Spoor and H. Zahn (Dtsch. Wollforschungsinst., Tech. Hochsch., Aachen, Germany). *Z. anal. Chem.*, 1959, **168** (3), 190–195.—The oligomers of caprolactam (obtained in 10% yield by polymerisation at 250°) are separated paper-chromatographically in aq. *sec.*-butyl alcohol - formic acid - water (15:3:2) after reduction by means of $LiAlH_4$ to the corresponding cyclic amines. The separation of the dimer and trimer is difficult. The amine spots are determined (to within ±15%) by chlorination to the chloroamines, which, after removal of the excess of Cl, liberate iodine (assayed colorimetrically at 420 mμ) from 1% KI in *N* acetic acid. It is essential to treat all the paper strips of one determination identically. The method is directly applicable to closely related members of a series. J. P. STERN

1100. **Determining the bound styrene in styrene - butadiene copolymers.** C. L. Hilton (Res. Center, U.S. Rubber Co., Wayne, N.J., U.S.A.). *Rubb. Age, N.Y.*, 1959, **85**, 783–785.—Refractive index, i.r. absorption and u.v. absorption methods are commonly used for this determination, but for accurate results the copolymer must be separated from all other ingredients and losses of polymer will result; the present method avoids several ether extractions. The sample is boiled for at least 16 hr. with conc. HNO_3. The reaction product is diluted, made alkaline, and then adjusted to volume. An aliquot of the solution is further diluted and the extinction of this solution, essentially due to Na *p*-nitrobenzoate produced from the polymerised styrene, is determined at 275, 285 and 300 mμ. The bound styrene is calculated from these figures. J. H.

1101. **Estimation of Phenidone [1-phenylpyrazolid-3-one] in used developers.** G. I. P. Levenson and M. G. Rumens (Res. Lab., Kodak Ltd., Wealdstone, Middx., England). *J. Photogr. Sci.*, 1959, **7** (2), 38–40.—A method for the determination of 1-phenylpyrazolid-3-one (**I**) in used **I** - quinol developers is described. The soln. (100 ml ≡ 0·15 to 0·35 g of **I**) is extracted twice with 50 ml of $CHCl_3$ at pH ≃8·3, and the $CHCl_3$ extracts are added to 400 ml of *N* H_2SO_4; 0·05 *N* iodine in $CHCl_3$ (12 ml) is added and back-titrated with 0·05 *N* $Na_2S_2O_3$, with starch as indicator. Provided that at least 50% excess of iodine is used, the reaction is nearly stoicheiometric and secondary reactions are prevented. G. H. EMMOTT

1102. **Microchemical and spectrographic analysis of the residual materials in developed photographic layers.** K. F. Eremenko and A. M. Berlin. *Trudy Vses. Nauch.-Issled. Kinofoto Inst.*, 1958, [3 (26)], 11–16; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,602.—The gelatin layers of colour positives on multi-layer plates are studied. Film frames were treated with water (20 ml) for 24 hr. and $S_2O_3^{2-}$ determined in the resulting extract by means of NaN_3 and iodine. A large amount of $Na_2S_2O_3$ was found in positives treated by a rapid method, but was absent in those obtained by a single process; SO_3^{2-}, $Fe(CN)_6^{3-}$ and $Fe(CN)_6^{4-}$ were not detected in the extracts. After obtaining the extract the gelatin layer was hydrolysed with alkali or a proteolytic enzyme; the hydrolysate was separated from the base and treated with $CHCl_3$ to remove the dyestuffs. The remaining soln. was filtered under pressure, and the pasty residue analysed. Testing the residue with soln. of $Pb(NO_3)_2$, $FeCl_3$, $FeSO_4$ and NH_4SCN showed the absence of $Fe(CN)_6^{3-}$, $Fe(CN)_6^{4-}$ and Fe^{3+}. Acid treatment of the dry residue obtained by evaporation of the aq. extracts showed the presence of Na^+ and Ag^+; this indicates the presence in the photographic layers of soluble complexes of Na and Ag. The ash obtained by ignition of the photographic layers was qual. analysed spectrographically, confirming the presence of Ag and Na. C. D. KOPKIN

1103. **Gravimetric determination of cellulose acetate in double-base propellents.** A. N. Fletcher, R. H. Pierson and E. St. Clair Gantz (U.S. Naval Ordnance Test Sta., China Lake, Calif.). *Anal. Chem.*, 1959, **31** (7), 1224–1226.—The analysis of a propellent mixture of cellulose acetate (**I**) and nitrate, glycerol triacetate and trinitrate, and lead salts is difficult. Alkaline hydrolysis converts **I** into cellulose (**II**) and the nitrate yields water-soluble products. The **II** is then determined gravimetrically, corrections being applied for the insol. matter originally present in the **I**. The accuracy for 5% of **I** is within ±1%. The operator time for 6 determinations is ≃2 hr. per sample. *Procedure*—Hydrolyse the sample (4 g of 20-mesh) with 250 ml of 0·45 ± 0·05 *N* KOH [in diethyl ether - ethanol - H_2O (49:46:5)] for 15 hr., acidify to phenolphthalein, and filter through an Alundum extraction thimble, washing all **I** into the thimble with 50% acetic acid at 40° to 60°, and then with acetone. Dry at 100° ± 5° for 3 hr. at ≯40 torr and weigh. J. P. STERN

1104. **Ethanediol antifreeze. Type A. Triethanolammonium orthophosphate and sodium mercaptobenzothiazole inhibited.** British Standards Institution (2 Park Street, London, W.1). B.S. 3150:1959, 16 pp.—Methods are described for the determination of freezing-point, pH, sodium, phosphoric acid and mercaptobenzothiazole.

1105. **Ethanediol antifreeze. Type B. Sodium benzoate and sodium nitrite inhibited.** British Standards Institution (2 Park Street, London, W.1). B.S. 3151:1959, 13 pp.—A specification is given for the sodium benzoate, and methods are described for the determination of freezing-point, pH, benzoate and nitrite.

1106. **Ethanediol antifreeze. Type C. Sodium tetraborate inhibited.** British Standards Institution (2 Park Street, London, W.1). B.S. 3152:1959, 12 pp.—Methods are given for the determination of freezing-point, pH and borate.

See also Abstracts—**851**, Na tetraphenylboron for analysis of organic bases. **854**, Indicators for oxalate titrations with ceric perchlorate. **859**, Use of dielectric constants in analyses. **870**, Moisture in anthracite, lignite, etc. **1140**, Cysteic acid in wool. **1200**, Chromatography of carboxylic acids. **1229**, Determination of S and Cl^- in methacrylates.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1107. **Simplified flame-spectrophotometric method for estimation of magnesium in serum.** D. D. van Fossan, E. E. Baird and G. S. Tekell (Clin. Lab., Dept. of Path., Univ. of Texas, Galveston). *Amer. J. Clin. Path.*, 1959, **31** (4), 368–370.—Serum is deproteinised with trichloroacetic acid, and acetone is added up to 80% (v/v). After centrifuging, the luminosity of the supernatant liquid is measured with a flame photometer at 285·2 mμ. Standard soln. are also measured. Acetone increases the sensitivity and the flame stability. The method compares favourably with the Titan yellow method for accuracy, reproducibility and specificity, and is considerably more rapid. P. NICHOLLS

1108. **Spectrochemical determination of magnesium, chromium, nickel, copper and zinc in human plasma and red cells.** L. M. Paixao and J. H. Yoe (Pratt Trace Anal. Lab., Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Clin. Chim. Acta*, 1959, **4** (4), 507–514.—A spectrographic method is described, and the results are compared with those previously reported in the literature. H. F. W. KIRKPATRICK

1109. **The complexometric determination of calcium in serum.** M. Velek and M. Valentová (Centr. Psychiatr. Lab., Prague). *Čas. Lék. Čes.*, 1959, **98** (32–33), 1025–1028.—The following method is recommended. *Procedure*—Mix the sample (1 ml) with ammonium oxalate soln. (1%) (1 ml); after 15 min. separate the pptd. Ca oxalate in a centrifuge, dissolve it in *N* HCl (1 ml) and transfer to a titration flask with H_2O (50 ml). Add *N* NaOH (1·5 ml), a small amount of Fluorexone (calcein) (1:100 with NaCl) as indicator, and titrate with 0·001 *M* EDTA (disodium salt) till the yellow fluorescence changes to yellow - orange. J. ZÝKA

1110. **Use of Metomega Chrome blue BBL as a metal indicator and its application for the determination of calcium and magnesium in serum.** A. A. Abd El Raheem and M. M. Dokhana (Public Health Lab., Cairo, Egypt). *Z. anal. Chem.*, 1959, **168** (3), 165–172 (in English).—Metomega Chrome blue BBL (C.I. Mordant Blue 26) (**I**) in 10% diethylamine soln. changes to red, pink or violet with Ba (8·2), Cd (0·67), Ce^{3+} (4·2), Ca (0·16), Co (0·07), Cu (0·17), Mg (0·085), Mn (0·1), Ni (0·13), Pb (1·4) or Zn (0·3 μg), and can be used as an indicator for chelometric titrations with EDTA. Thus Mg, Mn, Ni and Zn are titrated at pH 10 (Ni at 80° and Mg in the presence of ascorbic acid) and Ca in aq. NH_3 (pH 11) or diethylamine (pH ≃ 13). Calcium and Mg can be determined in the presence of each other by stepwise titration, first at pH 11 to 13 and then at pH 10; in serum, Ca (10 mg) and Mg (2 to 3 mg) are thus determined with an accuracy within ±0·5 and ±2%, respectively. For the detection of all these cations at the limit of sensitivity a fenugreek mucilage is used. *Procedure for serum*—Mix serum (1 ml) with 20% trichloroacetic acid (1 ml), set aside for 10 min., then centrifuge; treat 1 ml of the clear soln. with aq. NH_3 (1 drop), 10% diethylamine soln. (5 ml) and KCN (a small crystal). Add 0·1% aq. **I** as indicator and titrate with 0·001 *M* EDTA (disodium salt) (**II**) to a blue colour for the determination of Ca. Boil the soln. for 2 min., add HCl (1 drop), 5 ml of twice-distilled H_2O, and 2 ml of buffer (pH 10) (13·5 g of NH_4Cl and 88 ml of conc. aq. NH_3, diluted with H_2O to 250 ml) and titrate with **II** to a blue end-point for the determination of Mg. J. P. STERN

1111. **Micro-determination of traces of lead in blood or other biological media.** W. Haerdi and D. Monnier (Lab. de Microchim., Univ., Geneva). *Mitt. Lebensmitt. Hyg., Bern*, 1959, **50** (4), 243–257.—A polarographic method is described in which the sample (3 to 10 ml of blood) is wet-ashed and the Pb is extracted with dithizone and then determined polarographically. The sensitivity is 1 μg and the accuracy is ±10%. I. DICKINSON

1112. **Phosphorus metabolism of erythrocytes. I. Analysis and distribution of acid-soluble phosphorus in normal human erythrocytes.** P. Cartier, J. Chedru and P. Baudequin (Lab. Michel Polonovski, École de Médecine, Rouen). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 525–536.—The erythrocytes are labelled *in vitro* with trace amounts of $^{32}PO_4^{3-}$, and all subsequent analyses are performed on a $HClO_4$ soln. of the haemolysed labelled cells. Orthophosphate is determined directly by the method of Martin and Doty (*Brit. Abstr. C*, 1950, 48) in which it is extracted and determined as molybdophosphate in an *iso*butyl alcohol - benzene mixture. The separation of the nucleotide and phosphorus ester fractions is achieved by passage of the acid soln. through a mixed column of charcoal - kieselguhr (1:1); the nucleotides remain on the column, the percolate contains the phosphorus esters. The ester fraction is determined as the difference between the total phosphorus content of the percolate, measured by the method of Fiske and Subbarow (*J. Biol. Chem.*, 1925, **66**, 375), and its orthophosphate content. To determine the nucleotides, they are eluted from the column with 10% aq. pyridine, and separated by descending paper chromatography with *iso*butyric acid - water - aq. NH_3 ($d = 0·9$) (66:33:1). The separated compounds are located under u.v. light and then determined spectrophotometrically after elution with dil. HCl. The specific activity of each compound is measured at all stages of this procedure. The methods used are discussed, and the reproducibility of each is given for an average of 80 determinations. Values for the various compounds determined (*i.e.*, orthophosphate, phosphorus esters, and adenosine mono-, di- and tri-phosphoric acids) are reported for 20 normal adults, and are compared with those obtained by other workers. D. B. PALMER

1113. **Micro-determination of fluoride in urine.** G. J. Mulder (Farm. Lab. der Rijksuniv., Utrecht, Netherlands). *Pharm. Weekbl.*, 1959, **94** (10), 329–375.—The method is based on the decrease in colour intensity of a soln. of Zr - Eriochrome cyanine R (C.I. Mordant Blue 3) on the addition of F^-, due to the formation of ZrF^{3+}, and the measurement of this decrease by differential spectrophotometry (*cf.* Megregian, *Anal. Abstr.*, 1954, **1**, 2676). *Procedure*—Transfer 100 ml of urine, containing ≯150 μg of F^-, to a platinum dish and add 5 ml of Mg acetate soln. (400 g of Mg acetate tetrahydrate per litre) and make just alkaline to phenol red with 4 *N* NaOH. Evaporate to dryness and heat the residue at 600° for 2 hr. Steam-distil the residue with 10 ml of water and 20 ml of H_2SO_4 (35%) (*cf.* Huckabay *et al.*, *Ind. Eng. Chem., Anal. Ed.*,

1947, **19**, 154), collecting 200 ml of distillate in 50 ml of water to which have been added 6·5 ml of 4 *N* NaOH and 2 drops of phenolphthalein soln. Neutralise the distillate with 4 *N* HCl to 2:6-dinitrophenol indicator and add 10% aq. NH_3 till the soln. is just pink. Evaporate at $\simeq 80°$ to $\simeq 50$ ml, then add 50 ml of Eriochrome cyanine R soln. (0·36%), make up to 100 ml and add 10 ml of $ZrOCl_2$ - HCl soln. (dissolve 1 g of $ZrOCl_2.8H_2O$ in 500 ml of water, and to a 25-ml aliquot add 60 ml of conc. HCl and make up to 200 ml). Measure the extinction of this soln. at 555 mμ against a soln. prepared by carrying 50 ml of a soln. of 150 μg of F^- and 1·5 g of NaCl through the last part of the procedure. Determine the F^- with the aid of a calibration graph prepared from soln. containing known amounts of F^- (< 150 μg). The standard deviation of 16 results on 3 samples is ±3·7 μg of F^- per 100 ml.

M. J. MAURICE

1114. **Determination of chloride in blood serum, plasma or other biological fluids by a new rapid precision method.** H. V. Malmstadt and J. D. Winefordner (Dept. of Chem., Univ. of Illinois, Urbana, U.S.A.). *Clin. Chem.*, 1959, **5** (4), 284–286.—A rapid potentiometric method for small samples is described. The relative error is < 0·5%.

H. F. W. KIRKPATRICK

1115. **Determination of iron in plasma.** R. Willocx (Clin. Méd., Univ. Louvain). *Arch. Int. Pharmacodyn.*, 1958, **117**, 100–113.—To serum or plasma (2 ml) was added 6 *N* HCl (2 ml) followed, after 10 min., by 20% trichloroacetic acid (2 ml). The mixture was heated at 70° for 10 min. To the filtrate (2 ml) were added 1 drop of *p*-nitrophenol, conc. aq. NH_3 until a yellow colour was produced and then *N* HCl until the soln. was decolorised (pH 1·7 to 2·1). Three drops each of quinol and 1:10-phenanthroline were added, the soln. was set aside for 8 hr. in the dark, the vol. was adjusted to 5 ml and the extinction was read at 509 mμ; similar procedures were followed without 1:10-phenanthroline and with water with and without the dye. The mean value of 250 determinations in plasma taken from women was 86·7 μg per 100 ml, and of 100 determinations in men 121·3 μg per 100 ml; 50 to 180 μg was considered normal.

NUTR. ABSTR. REV.

1116. **Method for the rapid detection of small concentrations of organic bases in urine.** P. J. Morgan (Dept. of Pharmacol., Univ. of Melbourne, Australia). *Analyst*, 1959, **84**, 418–422.—In the general procedure for detecting morphine and other typical alkaloids, the urine adjusted to pH 9 with NaOH is extracted with $CHCl_3$ and the extract is filtered. A strip of filter-paper spotted with drops of 0·25 and 0·5 *N* H_2SO_4 is immersed in the extract under specified conditions, and a stream of O-free N (prep. described) is passed through the liquid. The dried strip is immersed in NH_3 vapour and the mixture of sulphates of the bases and $(NH_4)_2SO_4$ is submitted to ascending paper chromatography with a suitable solvent [the organic-rich phase of a mixture of *n*-butanol, conc. HCl and water (5:1:2) for typical alkaloids, and *iso*butyl methyl ketone, acetic acid and water (4:2:1) for such compounds as amphetamine and ephedrine]. For detection, the paper is sprayed with a mixture of 5% chloroplatinic acid, 4% KI soln. and water (2:25:20) for the alkaloids, and dil. ethanolic bromocresol green for the amines. Examples of the use of the method are given.

A. O. JONES

1117. **Determination of glutethimide in whole blood.** E. Algeri (Dept. of Legal Med., Harvard Univ. Med. Sch., Boston, Mass.). *Amer. J. Clin. Path.*, 1959, **31** (5), 412–413.—The method is based on the measurement of the extinction at 235 mμ of glutethimide (**I**) in ethanolic KOH soln. Blood is extracted with ethyl acetate and the solvent layer is then washed successively with dil. NaOH soln. and HCl. After filtration, an aliquot of the organic layer is boiled with charcoal and filtered, and the filtrate is evaporated to dryness. The residue is dissolved in 95% ethanol and KOH is added. The extinction of the resulting soln. is measured at 235 mμ by the method of Goldbaum *et al.* (*Fed. Proc.*, 1957, **16**, 300). The recovery of **I** added to blood is 100 ± 5% in the range 5 to 50 μg per ml. The method is suitable for the determination of **I** in blood after the administration of therapeutic doses.

P. NICHOLLS

1118. **Quantitative determination of glucose in urine.** M. Trząski and Z. Lisowski. *Farm. Polska*, 1958, **14**, 345–346.—The reduction of the complex salt of Cu and Na lactate was applied to the quant. determination of glucose in urine. The reagent is prepared by mixing equal parts of (*i*) a soln. of 17·3 g of $CuSO_4.5H_2O$ in 500 ml of water and (*ii*) 85% lactic acid (70·0 g), NaOH (76·6 g), Na_2CO_3 (10·0 g), $K_4Fe(CN)_6$ (3·2 g) and KSCN (5·0 g) dissolved, in this order, in 500 ml of water; 1 ml of the reagent ≡ 4 mg of glucose. For the determination, urine is added dropwise from a micro-burette to 2 ml of the reagent, with boiling after each addition. The reagent discolours gradually until 1 drop of urine causes the appearance of a yellow-orange coloration, indicating the complete reduction of the reagent. The concn. of glucose in the urine is calculated from the amount of urine added. For samples containing > 1% of glucose, the urine is diluted with water for greater accuracy. The results agree with those obtained by polarimetric methods to within ±1%.

CHEM. ABSTR.

1119. **Radioactive sub-micro determination of urea by the method of Velluz and Bertin, with labelled [9-^{14}C]xanthhydrol. Biological applications.** M. Herbain and D. Bertin (Serv. Rech. Roussel-Uclaf, Romainville, Seine, France). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 621–632.—The reaction between xanthhydrol and urea to give dixanthylurea is used for the determination of urea in biological media. This method is a modification of that due to Velluz *et al.* (*Compt. Rend.*, 1958, **246**, 3071), a methanolic soln. of xanthhydrol replacing the original acetic acid soln. The pptd. radioactive dixanthylurea is isolated and determined by direct counting. A study of the various physical and chemical factors affecting the method is made. It is shown that, over a range of 10 to 100 μg of urea, the reproducibility is ±5%. The application of the method to the determination of blood urea is described; as little as 0·1 ml of sample is required.

D. B. PALMER

1120. **Quantitative determination of sugars on paper chromatograms.** C. M. Wilson (New Jersey Agric. Expt. Stat., New Brunswick, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1199–1201.—Mixtures of glucose, galactose, arabinose, xylose and rhamnose, particularly in hydrolysates of plant-cell walls (50 mg), are separated chromatographically on paper and determined by eluting the spots, coloured with aniline hydrogen phthalate (**I**), by means of

ethanolic HCl and measuring the extinction at 390 mμ (for hexoses and rhamnose) and 360 mμ (for pentoses). A standard soln. is treated simultaneously on the same paper. The chromatogram is developed by three descending treatments with H_2O - *n*-butanol - pyridine (3:6:4). The absorption peaks are broad, and results on 100 μg of a sugar are reproducible to within ±2%. Variability is increased by the use of several papers. Uniformity of distribution of **I** is essential and is achieved by dipping the paper into a soln. of **I** in H_2O - *n*-butanol - diethyl ether (1:12:12).

J. P. STERN

1121. **Microscopic identification of microgram quantities of galacturonic acid and glucuronolactone. Direct synthesis of hydrazone derivatives by the solvent diffusion technique.** L. M. White and G. E. Secor (W. Utilization Res. and Devel. Div., U.S. Dept. of Agric., Albany, Calif.). *Anal. Chem.*, 1959, **31** (7), 1273–1274.—After separation from other sugars by descending paper chromatography with H_2O - ethyl acetate - pyridine (1:8:2) as solvent, washing, and elution with H_2O, glucuronic acid (10 to 15 μg) is identified microscopically as glucuronolactone 2:4-dinitrophenylhydrazone, and galacturonic acid (>2 μg) is identified microscopically as the 2:5-dichlorophenylhydrazone.

J. P. STERN

1122. **The structural carbohydrates of grasses.** R. Waite and A. R. N. Gorrod (Hannah Dairy Res. Inst., Kirkhill, Ayr, Scotland). *J. Sci. Food Agric.*, 1959, **10** (6), 308–317.—A detailed analysis of the structural carbohydrates of a number of grasses at different stages of growth is described. Holocellulose was prepared by extracting grass successively with ethanol - benzene (1:2), H_2O, 0·5% ammonium oxalate soln., and 1% pepsin soln. in 0·1 *N* HCl; the resulting fibre was delignified with chlorite - acetic acid reagent (Whistler *et al.*, *Arch. Biochem.*, 1948, **19**, 25). Hemicelluloses were removed by successive extraction with H_2O, 1% KOH soln. and *N* H_2SO_4; after hydrolysis of the filtrates and residue, galactose, glucose, arabinose and xylose were separated by paper chromatography and determined with periodate (Hirst and Jones, *J. Chem. Soc.*, 1949, 1659). Uronic acids were determined by acid digestion (Johannson *et al.*, *Svensk Papp.-Tidn.*, 1954, **57**, 41). Changes in composition with age and type of grass are discussed.

P. D. PARR-RICHARD

1123. **Comprehensive analysis of grasses.** R. Waite and A. R. N. Gorrod (Hannah Dairy Res. Inst., Kirkhill, Ayr, Scotland). *J. Sci. Food Agric.*, 1959, **10** (6), 317–326.—By successive extraction of grass, 97 to 98% of the dry matter could be accounted for and all but 1 to 3% of it identified. Extraction with ethanol - benzene (1:2), evaporation of the solvent and partition of the residue between diethyl ether and H_2O yielded fats, waxes, phospholipids and free fatty acids. The aq. layer, combined with material obtained by a brief aq. extraction of the residual grass, contained polysaccharides, organic acids and non-protein nitrogen compounds. The grass was then extracted with 0·5% ammonium oxalate soln. to remove pectin, and by pepsin in 0·1 *N* HCl to remove residual protein. Delignification of the residue yielded holocellulose, which was in turn examined by successive extraction with hot H_2O, 1% KOH soln., *N* H_2SO_4 and 72% H_2SO_4 soln.

P. D. PARR-RICHARD

1124. **Spectrophotometric determination of quercetin.** L. E. Dowd (Central Res. and Development, Weyerhaeuser Timber Co., Longview, Wash., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1184–1187.—The method is based on the formation of a coloured complex with $AlCl_3$. The complex exhibits max. extinction at 430 mμ in 0·01 *M* $AlCl_3$ adjusted to pH 4·0 with K acetate, and Beer's law is followed over the concn. range from 2 to 15 μg of quercetin per ml. The standard deviation is ±0·4% and the 95% confidence limit for triplicate determinations is ±1%. Phlobaphenes and tannins present as impurities do not interfere.

K. A. PROCTOR

1125. **Microbiological method for detecting folic acid deficiency in man.** H. Baker, V. Herbert, O. Frank, I. Pasher, S. H. Hutner, L. R. Wasserman and H. Sobotka (Mt. Sinai Hosp., New York, U.S.A.). *Clin. Chem.*, 1959, **5** (4), 275–280.—*Lactobacillus casei* is used in the method described, and the results correlate directly with the clinical diagnosis.

H. F. W. KIRKPATRICK

1126. **Extraction of pigments from plant material.** V. H. Booth (Dunn Nutritional Lab., Univ. of Cambridge, England). *Analyst*, 1959, **84**, 464–465.—Small samples are weighed in beakers as previously described (Analytical Methods Committee, *Analyst*, 1950, **75**, 568). An equal wt. of quartz and a few mg of quinol are added, followed by ≃5 ml of acetone, and the material is ground in the beaker. Light petroleum (≃5 ml) is then added and, after ≃1 min., the supernatant liquid is decanted into a layer of light petroleum on a half-saturated soln. of $(NH_4)_2SO_4$ in a separator. The residue is ground again and extracted as before, the procedure being repeated until all the pigment has been extracted. The combined extracts are washed with water and the procedure is then as previously described (*loc. cit.*). The method has been used for the extraction of a variety of biological substances from plants.

A. O. JONES

1127. **Paper chromatography of lipids.** H. K. Mangold, J. L. Gellerman and H. Schlenk. *Fed. Proc.*, 1958, **17**, 268; *Ann. Rep. Hormel Inst.*, 1957–1958, 30–31.—A method for the almost complete analysis of mixtures by the exclusive use of paper-chromatographic procedures is reported; it involves the use of solvents which react with double bonds. The developing solvent consisted of peracetic acid or 30% H_2O_2 - acetic acid - water (2:15:3). Unsaturated lipids migrated as oxygenated compounds with R_F values (>0·8) greatly different from those of the original compounds. Saturated lipids were separated normally but free from superimposing unsaturated components. The validity of the method for quant. use was established with randomly labelled single-chain lipids. Chemical methods for detecting fatty acids become necessary when N-methylanilides are chromatographed. They can be separated in acidic solvents and in u.v. light are visible on chromatograms as blue spots. Similarly, direct physical detection and quant. measurement of the lipid components can be made with the use of [^{14}C]-methyl esters. Radioactive diazomethane was obtained from toluene-*p*-sulphonyl[^{14}C]methylnitrosamide, which was prepared from [^{14}C]methylamine (0·05 mC per mole) on the gram scale. Several samples can be esterified by reaction with the diazomethane obtained from 50 mg of the nitrosamide. Test mixtures of C_{18} acids were esterified,

chromatographed and scanned. The limit of error in quant. determinations was ±5%.

REV. CURR. LIT. PAINT COL. VARN.

1128. **Modified hydroxamic acid method for determining total esterified fatty acids in plasma.** D. M. Morgan and K. J. Kingsbury (Surgical Unit, St. Mary's Hospital, London). *Analyst*, 1959, **84**, 409–414.—The plasma (0·5 ml) is added with agitation to ≃8 ml of an ethanol - ether mixture (3:1), the mixture is brought to b.p. in a water bath, cooled, adjusted to 10 ml and filtered. A 4-ml aliquot of the filtrate is treated with 1 ml each of hydroxyammonium chloride soln. (3·5 g in 30 ml of water diluted to 100 ml with ethanol) and NaOH soln. (6% in 50% aq. ethanol), and set aside in a stoppered tube. After 30 min., 1 ml of HCl (25 ml of conc. HCl made up to 100 ml with 50% ethanol) is added and then 1 ml of $FeCl_3$ soln. (6 g of $FeCl_3.6H_2O$ in 10 ml of *N* HCl and 10 ml of water, diluted to 100 ml with ethanol). After 30 min. the extinction of the coloured complex of hydroxamic acid with Fe is measured at 515 mμ and referred to a calibration graph, which is linear. The working standard soln. contains 0·001 milli-equiv. of the appropriate esterified fatty acid. Glycerol trioleate was used in the investigation. The normal range of esterified fatty acids in 106 samples of normal human fasting plasma was found to be 5·94 to 18·9 (mean 11·58) milli-equiv. per litre.

A. O. JONES

1129. **Spectrofluorimetric estimation of adrenochrome in human plasma.** A. N. Payza and M. E. Mahon (Psychiatric Res. Unit, Univ. Hosp., Saskatoon, Canada). *Anal. Chem.*, 1959, **31** (7), 1170–1174.—In the specific spectrofluorimetric method described, the plasma is treated with Zn acetate in ascorbic acid soln. to convert the adrenochrome into the fluorescent adrenolutin. Over the concn. range 0·1 to 1 μg a linear fluorescence curve is obtained. The fluorescence is read at 405 mμ excitation and 500 mμ emission.

K. A. PROCTOR

1130. **Determination of plasma glutamine by high-voltage paper electrophoresis.** J. Puranen, A. L. Puranen and N. Hallman (Child. Clin., Univ. Helsinki). *Ann. Paediat. Fenn.*, 1958, **4**, 203–208.—Plasma was deproteinised by filtering under pressure through a dialysing membrane. The filtrate (1 ml) was applied to a column of Dowex-50 resin (Cl^- form), which was eluted with water and then with *N* aq. NH_3. The amino acids in the second eluate were dissolved in *iso*propyl alcohol and separated in 2·5 to 3 hr. by paper electrophoresis at 50 to 60 V per cm and ≃1 mA, with a cooling agent at ≃13° and a buffer of 0·75 *N* formic acid and *N* acetic acid (1:1). After being sprayed with ninhydrin and treated with $Cu(NO_3)_2$ in acetone, the glutamine spot was cut out, the colour was eluted with methanol and the extinction measured at 530 mμ. A standard soln. of glutamine was run with each sample. Glutamine in the plasma of two patients with coeliac disease increased by 67 and 126% after the administration of 350 mg of gliadin per kg of body-weight; in two normal children the values fell by 14 and 10%.

NUTR. ABSTR. REV.

1131. **Chromatographic separation and spectrophotometric determination of adenosine triphosphate and adenosine diphosphate from animal tissues.** L. Longinotti. *R. C. Ist. Sup. Sanit.*, 1958, **21**, 866–876.—Fresh animal tissue is triturated in 60% alcohol at 0°, and the suspension is centrifuged at 0°; an aliquot of the clear liquid is removed and dried *in vacuo* over P_2O_5 at 5° for 24 hr., and the residue is taken up in cold water. A fine line of the extract is applied in droplets to Whatman No. 1 filter-paper [washed with 0·2% EDTA (disodium salt) soln. of pH 8·5 and rinsed with water] 14 cm above its lower edge, and the paper is dried. The orthophosphates are separated from the stationary adenosine triphosphate (**I**) and adenosine diphosphate (**II**) by descending chromatography, with dry diethyl ether - 99% formic acid (3:2) as solvent. The paper is dried, and cut off 7 cm from the lower edge. The remaining paper is subjected to descending chromatography with a mixture of *iso*butyric acid - *N* aq. NH_3 (10:3). The strip is dried and the spots of **I** and **II** are revealed under u.v. light at 230 to 400 mμ. The spots are cut out and eluted separately for spectrophotometry at 260 mμ. Both **I** and **II** have a mol. absorption of 15×10^3. For analysis, the acid-soluble P is determined by $HClO_4$ extraction, and the acid-labile P is determined by hydrolysis in *N* HCl for 10 min. at 100°. Commercial samples of **I** and **II** so analysed gave these results for **I**, **II** and adenosine monophosphate (all as μg of adenine), and percentage of adenine found: **I**—13·6, 0·8, 0·0, 94; and **II**—1·6, 17·8, 0·7, 96. Up to 200 μg of **I** plus **II** and 50 μg of orthophosphate can be separated in this way.

CHEM. ABSTR.

1132. **Determination of amino acids in small samples of blood.** D. Lohmann (Univ., Leipzig, Germany). *Dtsch. Z. Verdau.- u. Stoffwechselkr.*, 1958, **18**, 95–104.—Mix the serum (2 to 3 ml) with 3 vol. of ethanol and remove the ppt. by centrifuging. Evaporate the soln. to dryness at 37°, treat the residue with anhyd. ethanol containing 2% of *N* HCl, centrifuge and evaporate to dryness again. Dissolve the residue in 0·1 *N* HCl, subject the soln. to electrophoresis for 7 hr. at 175 V and 49 mA, with acetic acid (5 ml), pyridine (50 ml) and water (to 500 ml) as solvent at a pH of 6. To identify the acid, neutral and alkaline fractions, cut a strip lengthwise and stain with ninhydrin. Cut fractions of neutral and acid amino acids from the main strip, elute each with water, evaporate to dryness, dissolve the residue in water (0·5 ml) and subject to paper chromatography with butanol - acetic acid - water (4:1:1) as solvent. Pass the dried strips quickly through a soln. of 0·5 g of ninhydrin in acetic acid (7 ml) and butanol satd. with water (93 ml), hang them in the air for 20 min. at 35° to 42°, then place them again in the ninhydrin soln. at 60°. Separate the amino acid zones, and extract each with methanol (4 ml) and 2 drops of a soln. comprised of saturated $Cu(NO_3)_2$ soln. (2 ml), 10% HNO_3 (0·2 ml) and 96% ethanol (100 ml). After 30 min. read the soln. in a Pulfrich photometer at 500 mμ comparing them with standard amino acid soln. Standard curves are prepared by chromatography of the amino acids as described above. The chromatographic separation of the basic amino acids in phenol satd. with water gave arginine, lysine and histidine. The quant. evaluation by chromatography was not satisfactory. To determine the total basic amino acids mix 0·5 ml of soln. with 1 ml of KCN reagent [0·01 *M* KCN (2 ml) mixed with ammonia-free pyridine to make 100 ml] and 1 ml of a soln. of phenol (80 g) in absolute ammonia-free ethanol (20 ml), heat to 100°, add 0·5% ninhydrin in ethanol (0·2 ml), stopper the container and keep at 100° for 5 min.,

cool, add 60% ethanol (10 ml) and measure photometrically at 570 mμ. A standard curve is prepared with lysine in concn. from 10 to 60 μg. (The molar extinctions of arginine, lysine and histidine are practically equal.) Arginine was determined separately by the method of Sakaguchi (*J. Biochem. Japan*, 1950, **37**, 231; 1951, **38**, 91) with 8-hydroxyquinoline, and histidine by the method of Edlbacher *et al.* (*Hoppe-Seyl. Z.*, 1941, **270**, 158). CHEM. ABSTR.

1133. **Simple method for the detection of small amounts of α-amino acids.** R. Antoszewski and L. T. Antoszewska (Dept. of Plant Physiol., Univ. of Łódź, Poland). *Anal. Chim. Acta*, 1959, **20** (6), 595–596.—Absorbent paper is moistened with $CuCl_2.2H_2O$ soln. (0·03%), dried, dipped in $Na_2HPO_4.12H_2O$ soln. (0·15%) and washed with water. The test soln. is mixed with an equal vol. of a soln. that contains 57·21 g of $Na_2B_4O_7.10H_2O$ and 100 ml of *N* HCl in 2 litres. One drop of the mixture is applied to the paper, which is then washed with a drop of water, held over aq. NH_3 for 2 min. and treated with 0·1% ethanolic dithiooxamide soln. The presence of α-amino acids, down to 0·02 μg, in the test soln. is indicated by a white fleck on the dark-green paper. H. N. S.

1134. **Effect of saponin on the separation of amino acids by paper chromatography.** P. Ebrey (Pediatric Dept., Univ. of Szeged, Hungary). *Chemist Analyst*, 1959, **48** (2), 36.—In the paper chromatography of amino acids, the interaction of carboxyl and amino groups does not occur if the chromatogram is developed with phenol satd. with 20% aq. saponin soln. instead of water. After treatment with ninhydrin, the amino acids appear as discrete compact spots, which fade more slowly than those developed with phenol - water. S. M. MARSH

1135. **Improved solvent combination for amino-acid chromatography.** C. H. Bowden (Dept. of Chem. Path., Westminster Med. Sch., London, England). *Clin. Chim. Acta*, 1959, **4** (4), 539–544.—In this two-dimensional method the first run is made with phenol - water (4:1), adding 0·8 ml of aq. NH_3 (0·88) and 0·1 g of NaCN to the tank at the beginning of the run. The second run is made with ethyl methyl ketone - di*cyclo*hexylamine - *n*-butanol - water (10:2:10:5). The advantages claimed are that the solvent system is more mobile and pleasant to use, and gives a more favourable distribution of the important amino acids and greater colour differentiation. No preliminary distillation of the solvents is necessary, and satisfactory chromatograms are produced over a wide range of laboratory temp.

H. F. W. KIRKPATRICK

1136. **Rapid quantitative determination of amino acids by high-temperature paper chromatography.** J. B. Himes and L. D. Metcalfe (Armour & Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1192–1194.—The individual amino acid contents of mixtures or protein hydrolysates can be resolved in 2 hr. in a horizontal paper-chromatographic system at 60° with a solvent mixture of ethyl methyl ketone - propionic acid - water (15:5:6). The spots are developed with ninhydrin and the colour intensities read in a densitometer.

K. A. PROCTOR

1137. **Paper-chromatographic determination of arginine by the Sakaguchi reaction.** K. R. Bhattacharya (Indian Inst., Calcutta). *Sci. & Cult.*, 1958, **24**, 179–180.—The soln. (5 μl) is applied to Whatman No. 1 filter-paper and developed in *sec.*-butyl alcohol - pyridine - water - diethylamine by the ascending technique. The part of the paper containing the arginine is cut out, dipped in a mixture consisting of equal vol. of a 0·2% soln. of 1-naphthol in 95% ethanol containing 2% of urea, and 5% ethanolic KOH soln. After drying, the paper is dipped in a 0·75% soln. of Br in 5% aq. KOH soln. The dried arginine spots are eluted and measured colorimetrically. The colour intensity decreases with decreasing pH below pH 10·3. The method gives 100 ± 5% recovery with protein hydrolysates. CHEM. ABSTR.

1138. **Improved method for the determination of lanthionine.** M. X. Sullivan and C. F. Mijal (Georgetown Univ., Washington, D.C.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (6), 324–326.—Shake a soln. of the sample (containing 50 to 500 μg of lanthionine) in 0·1 *N* HCl (5 ml) with 5% NaCN soln. (1 ml), add cyanogen bromide soln. (prepared by adding 5% NaCN soln. dropwise to an aq. satd. soln. of Br until the product is colourless) (1 ml), stopper the test-tube and heat at 70° for 5 min. Cool, add a freshly prepared 0·5% aq. soln. of Na 1:2-naphthaquinone-4-sulphonate (**I**) (1 ml), shake for 40 sec., add a 10% soln. of sodium sulphite in 0·5 *N* NaOH (5 ml), stopper the tube and heat at 70° for 15 min. Cool, add a freshly prepared 2% soln. of $Na_2S_2O_4$ in 0·5 *N* NaOH (1 ml), shake and immediately read the extinction in a Klett - Summerson colorimeter fitted with a green filter (No. 54). To determine lanthionine in a wool hydrolysate, proceed as above, but correct by a reagent blank (omitting the sample) and a hydrolysate blank (omitting **I**). Methionine, thiodiglycollic acid and cystathionine give no colour. A. R. ROGERS

1139. **Photographic method for the detection of cysteine.** F. H. Schwarzenbach (Theodor Kocher Inst., Bern Univ., Switzerland). *Helv. Chim. Acta*, 1959, **42** (4), 1133–1141.—Drops of cysteine soln. on photographic film, after exposure and development, show as pale spots on a dark background. The effects of concn., pH, development conditions and the presence of other allied compounds are studied. Oxidising agents interfere. The method may be modified for use with filter-paper chromatograms or electropherograms before or after elution. Cystine and methionine do not interfere. The test soln. should be at pH 4 and the minimum quantity detectable is 0·24 μg. J. L. PROSSER

1140. **Quantitative determination of cysteic acid in protein hydrolysates. Rapid paper-electrophoretic method.** J. F. Diehl (Dept. of Biochem., Univ. of Arkansas, Little Rock, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1204–1206.—Low-voltage (approx. 250 V) paper electrophoresis at a constant current of 30 mA at pH 3·6 separates cysteic acid (**I**) from other amino acids in 2 hr. at a voltage gradient of 8 V per cm. The ninhydrin colour of **I** is determined either by direct photometry or by elution and colorimetry. The method is suitable for 2 to 20 μg of **I** per paper strip and is accurate to within ±3% (elution) or ±8% (direct photometry). The procedure is particularly useful for commercial wool samples, and takes about 5 hr. J. P. STERN

1141. **Spectrophotometric determination of amino acids. Alkaline copper salt method using cuprizone (bis*cyclo*hexanoneoxalyldihydrazone).** R. Borchers (Dept. of Biochem. and Nutrition, Univ. of

Nebraska, Lincoln, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1179–1180.—Amino acids may be determined on a 1-ml sample at 0·1 to 1·0 m*M* concn. The presence of non-amino interfering substances can be checked by prior treatment of a sample with HNO_3 to destroy the amino groups. The average recovery of alanine added to blood samples was 95·6%. K. A. PROCTOR

1142. **Spectrophotometric determination of dithiocarboxyaminocarboxylic acids.** Z. Mádlo and Z. Peizker (Prague XII, Lužická 18). *Pracovní Lékařství*, 1958, **10** (5), 411–413.—The method is based on the formation of a red product after treatment with N-ethylmaleimide (**I**) in an alkaline cyanide medium. *Procedure for N-dithiocarboxyalanine* (**II**)—To 1 ml of the soln. of **II** (7 to 100 μg) add 0·1 *M* aq. **I** (1 ml) and an alkaline soln. of KCN (5 g of KCN and 5 g of Na_2CO_3 dissolved in 100 ml of 25% ethanol) (0·2 ml). After 30 min. extract the coloured product with *n*-butanol (5 ml) and separate the organic layer. Repeat the extraction with 3 ml of *n*-butanol. Combine the extracts, dilute with ethanol to 10 ml and set aside for 15 min. Measure the extinction at 510 mμ. The mean error is ±7%. J. ZÝKA

1143. **Note on standardisation curves for γ-globulin determinations.** J. Fraenkel-Conrat (Bruce-Lyon Mem. Res. Lab., Children's Hosp. of the East Bay, Oakland, Calif., U.S.A.). *Clin. Chem.*, 1959, **5** (4), 281–283.—Two methods are described for correlating the wt. (g) of γ-globulin per 100 ml of serum with the extinction instead of expressing it in units, as in the turbidimetric method of Huerga and Popper (*J. Lab. Clin. Med.*, 1950, **35**, 459). H. F. W. KIRKPATRICK

1144. **Agar paper electrophoresis of A and F haemoglobins.** F. Rappaport and M. Rabinovitz (Beilinson Hosp., Petah-Tiqvah, Israel). *Clin. Chim. Acta*, 1959, **4** (4), 535–538.—A layer of 2% agar in buffer soln. (pH 5·7 to 5·9) (7 g of Na_2HPO_4 and 3·4 g of cryst. citric acid dissolved in H_2O to 1 litre) supported on a glass plate covered with filter-paper is used. Light green SF (C.I. Acid Green 5) (0·8 g of dye and 15 g of sulphosalicylic acid in 500 ml of H_2O) is used for staining (30 min.), followed by washing with 2% acetic acid until almost decolorised. The haemoglobins are well separated, haemoglobin A remaining near the point of application. H. F. W. KIRKPATRICK

1145. **New method of quantitative determination of serum proteins separated by paper electrophoresis.** W. Mejbaum-Katzenellenbogen and W. M. Dobryszycka (Dept. of Biochem., Wroclaw Univ. Sch. of Med., Poland). *Clin. Chim. Acta*, 1959, **4** (4), 515–522 (in English).—A reagent suitable for simultaneously fixing and dyeing the proteins on paper is prepared by dissolving 50 mg of bromophenol blue in 3 to 4 ml of ethanol and diluting to 100 ml with tannin reagent, prepared by dissolving 2 ml of phenol in 98 ml of *N* HCl at 80°, adding 10 g of tannic acid and keeping the soln. warm until dissolved, filtering if turbid. The moist or dry paper strips (drying for 10 min. at 90° is optional) are placed in this staining medium for 10 min., washed thoroughly with H_2O for 5 min., and dried between sheets of filter-paper at a temp. $<40°$. The strips are cut to separate the fractions and these are eluted in test-tubes with suitable amounts of 0·1 *N* NaOH to give a protein concn. within the range 10 to 100 μg per ml of eluate. The protein in each soln. is then determined turbidimetrically as follows. The soln. (1 ml) is placed in a water bath at 30° ± 1° for 5 min., 1 ml of tannin reagent, prewarmed to 30°, is added and mixed. After exactly 10 min., 2 ml of a 0·1% aq. soln. of acacia gum is added, the tube is cooled immediately and the turbidity is measured with a red filter against a reagent blank. Standard protein - turbidity curves prepared with serum, lysozyme or gelatin are linear for concn. up to 100 μg per ml of test soln. Whatman No. 1 paper and 0·02 ml of serum are used for the electrophoresis. H. F. W. KIRKPATRICK

1146. **Simplified rapid method for the fixation of paper electropherograms.** H. V. Connerty, A. R. Briggs and E. H. Eaton, jun. (Veterans Admin. Hosp., Wilmington, Del., U.S.A.). *Amer. J. Clin. Path.*, 1958, **30**, 343–344.—The serum proteins are separated by electrophoresis and the moist paper strips are placed in a soln. of ethanol and diethyl ether, which coagulates and denatures the proteins. The strips are then placed in a collodion soln., after which they are allowed to dry in air, and are then stained with a 0·1% methanolic soln. of bromophenol blue. The stain is intensified by placing the strips in an atmosphere of NH_3. The method is simpler and requires less time than other commonly used methods, and avoids the use of heat. CHEM. ABSTR.

1147. **Study of the ferric chloride method for the determination of total cholesterol and cholesterol esters.** N. Chiamori and R. J. Henry (Bio-Science Res. Foundn., Los Angeles, Calif., U.S.A.). *Amer. J. Clin. Path.*, 1959, **31** (4), 305–309.—In this study the determination of total cholesterol and cholesterol esters in serum by pptn. with ferric chloride - acetic acid, followed by colour development with H_2SO_4, is found to be simpler and more sensitive and reproducible than methods previously described. Interference by any bromide present can be eliminated by passage through an ion-exchange resin. P. NICHOLLS

1148. **Determination of serum α- and β-lipoprotein cholesterol.** N. Crawford (Surg. Res. Unit, Queen Elizabeth Hosp., Birmingham, England). *Clin. Chim. Acta*, 1959, **4** (4), 494–502.—Electrophoresis of the serum is carried out on triplicate strips. One is then stained with Sudan black B (C.I. Solvent Black 3) and the areas corresponding to the α- and β-lipoproteins are cut out from the remaining two. Cholesterol is extracted with $CHCl_3$ - methanol (2:1), the solvent is removed, and the determination is completed by the $FeCl_3$ method. H. F. W. KIRKPATRICK

1149. **Comparative determination of [urinary] hydroxylipids, and of the different types of urinary steroids.** S. H. Weinmann and M. F. Jayle (Lab. Chim. Biol., Fac. de Méd., Paris). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 487–492.—The levels of formaldehyde-producing steroids, acetaldehyde-producing steroids, 17-oxosteroids, pregnanediol and hydroxylipids are determined in enzyme hydrolysates of urine. Values are reported for children and adults of both sexes, for women at various stages of the menstrual cycle, for pregnant women, and for subjects with well-defined adrenocortical disease. It is shown that the determination of urinary hydroxylipids is valueless as a measure of the total urinary hydroxysteroids, owing to its lack of specificity when applied to crude urine extracts. However, this assay is of value when

applied to pure steroid fractions, and in certain physiological and pathological states, when it will detect compounds not determined by the usual techniques. D. B. PALMER

1150. **Factors in the colorimetric estimation of 17-oxosteroids in urine.** J. B. Sheath (Diabetic and Metab. Unit, Alfred Hosp., Melbourne). *Aust. J. Exp. Biol. Med. Sci.*, 1959, **37** (2), 133–145.—Correction formulae for interfering chromogens are not satisfactory; elimination of interference by the addition of formaldehyde before acid hydrolysis of the sample is preferable. The optimum conditions for colour development in the Zimmermann *m*-dinitrobenzene reaction are defined as 45 min. at 25°, and methanolic KOH is substituted for ethanolic KOH as it is more stable. The recommended routine method consists in the addition of 0·15 ml of 40% formaldehyde soln. to 50 ml of urine, and acid hydrolysis with 15% of conc. HCl at 100° for 15 min., followed by extraction with diethyl ether. The colour reaction is carried out as described above, and the colour is read at 520 mμ against a reagent blank and compared with standards. No correction is necessary. H. F. W. KIRKPATRICK

1151. **Chromatography of urinary ketosteroids.** J. B. Sheath (Diabetic and Metab. Unit, Alfred Hosp., Melbourne). *Aust. J. Exp. Biol. Med. Sci.*, 1959, **37** (2), 147–152.—Urine is hydrolysed with β-glucuronidase and extracted with diethyl ether; the residual urine is hydrolysed with acid and again extracted with ether. The combined ether extracts are dried, and the ketonic fraction is separated with Girard's reagent T, dissolved in ethanol and chromatographed on Whatman No. 2 paper, with the descending technique. The solvent system is light petroleum - methanol - H_2O (2:1:1), the aq. layer being the stationary phase and the organic layer the mobile phase. The ketosteroid spots are revealed with alkaline *m*-dinitrobenzene (Savard, *J. Biol. Chem.*, 1953, **202**, 457). R_F values of pure ketosteroids and of compounds recovered from urines are tabulated. The identification of the recovered compounds is discussed. H. F. W. KIRKPATRICK

1152. **Determination of cortic[oster]oids in human urine. A review and comparison of existing methods.** R. Raptopoulo, H. Staudinger and L. Weissbecker (Zentrallab. d. Stadt. Krankenanst., Mannheim, Germany). *Clin. Chim. Acta*, 1959, **4** (4), 463–483.—An investigation of various methods is described. H. F. W. KIRKPATRICK

1153. **Investigation of the separation of synthetic steroid esters by paper electrophoresis.** O. J. Schmid, A. Oriol-Bosch and K. D. Voigt (Physiol. Chem. Inst., Univ.-Krankenhaus, Hamburg-Eppendorf, Germany). *Clin. Chim. Acta*, 1959, **4** (4), 599–605.—Eight synthetic steroid conjugates were used in tests with various pH values, buffer soln., points of application and voltages. The best results were obtained in separations at pH 3·5. H. F. W. KIRKPATRICK

1154. **Determination of cholinesterase activity in blood and organs by automatic titration.** J. Jensen-Holm, H. H. Lausen, K. Milthers and K. O. Møller (Copenhagen Univ., Denmark). *Acta Pharm. Tox., Kbh.*, 1959, **15** (4), 384–394.—The method described is based on the automatic titration of the acidity produced by the action of the cholinesterase in the sample on acetylcholine iodide as substrate. The pH is adjusted to 7·4 and automatically maintained at this value by titration with 0·1 *N* NaOH after the reaction has been started by the addition of the substrate. After deduction of the acidity produced by the non-enzymatic hydrolysis of substrate and the non-specific liberation of acid during titration, cholinesterase is calculated from the vol. of NaOH required and is expressed as μmoles of acid per ml per min. The errors of the method, and the colorimetric determination of acetylcholine by the ferric hydroxamate method, are discussed. W. H. C. SHAW

See also Abstracts—**894**, Determination of Ca in bones and teeth. **1194**, Pipette for deproteinising. **1208**, Electrophoresis of serum proteins.

Pharmaceutical analysis

1155. **Use of gas chromatography for determining water in pharmaceutical preparations.** D. A. Elvidge and K. A. Proctor (Standards Dept., Boots Pure Drug Co. Ltd., Beeston, Nottingham, England). *Analyst*, 1959, **84**, 461–463.—Standard soln. containing 0·5 to 2·0% of water and 2% of *n*-pentanol are prepared in acetone. The samples are shaken vigorously with acetone, diluted as necessary with acetone and 2% of *n*-pentanol is added. Standards and samples are examined by using the equipment previously described (Brealey *et al.*, *Ibid.*, 1959, **84**, 221), and a calibration graph is drawn by plotting the peak heights against water (%). The graph does not pass through the origin owing to the presence of a small amount of water in the acetone. The peak height is linearly related to the concn. of water, and the method has good reproducibility. Quoted results for a variety of pharmaceutical preparations agree with those obtained by the Dean and Stark method. The use of *n*-pentanol as an internal standard removes the difficulty of always transferring an exactly reproducible amount of sample to the column. A. O. JONES

1156. **Electrodialytic isolation of alkaloids from plant materials for quantitative analysis.** Z. Drabent and Z. Podeszewski (Dept. of Gen. Chem., Agric. Coll., Olsztyn, Poland). *Acta Polon. Pharm.*, 1959, **16** (4), 299–307.—The complete extraction of alkaloids from plant raw materials (nux vomica and cinchona bark) was accomplished in 15 to 20 min. by the electrodialysis of soln. acidified with acetic acid at 580 V d.c.; the extracted alkaloids proved suitable for direct analysis without further purification. The method is recommended for the quant. determination of the alkaloid content of the materials named. W. B. MIASKOWSKI

1157. **Applications of paper partition chromatography to the separation and identification of alkaloids in toxicological analysis.** C. Dorier, J. Dauphin, J.-C. Redon and J.-A. Berger (Lab. de Chim. Pharm. et Tox., Fac. de Méd. et de Pharm., Clermond-Ferrand, France). *Bull. Soc. Chim. France*, 1959, (4), 620–624.—The ethanolic extract of human viscera (obtained by a method described) containing added alkaloids is evaporated almost to dryness, re-dissolved in ethanol, centrifuged and evaporated to dryness; the residue is dissolved in water, then acidified and extracted with diethyl ether. The soln. is adjusted to pH 8 with Na_2CO_3 and re-extracted with ether. The ether soln. are evaporated and taken up in 0·5 ml of ethanol for chromatography. The paper chromatography is carried

out by descending or circular techniques on paper treated with phosphate or phosphate - tartaric acid buffer (pH 3, 5, 6·3, or 7·5) with a soln. of *n*-butanol saturated with the same buffer. The spots are revealed by a mixture of 10% KI soln. (45 ml), 5% H_2PtCl_4 soln. (5 ml) and water (100 ml). Results with visceral extracts containing added alkaloids (cocaine, strychnine, nicotine, brucine, morphine, hyoscine, sparteine and quinine) are described, and the viscera of a dog poisoned with strychnine were successfully examined. It is suggested that a dose of 2 mg administered to a 15-kg dog (much less than the lethal dose) could be detected.

E. J. H. BIRCH

1158. **Determination of rotenone in pharmaceuticals.** J. T. Bryan and W. R. Lincoln (Veterinary Res. Lab., The Upjohn Co., Kalamazoo, Mich.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (6), 330–332.—Dilute the sample (an emulsion-type lotion containing $\simeq$1 mg of rotenone) to 5 ml with H_2O and extract with $CHCl_3$ (3 × 10 ml). Dilute the combined $CHCl_3$ layers to 100 ml with $CHCl_3$ and set aside in contact with anhyd. Na_2SO_4 for 1 hr. Evaporate to dryness a 10-ml aliquot of the dried soln. and set aside for 1 hr. *in vacuo* over $CaCl_2$. Dissolve the residue in acetone (2 ml), place in a bath of water at 28° ± 0·5° for 5 min., add 2 ml of reagent (mix 7 vol. of a 0·1% soln. of $NaNO_2$ in 95% ethanol with 1 vol. of 40% KOH soln.) and allow to react for 5 min. Cool in ice for 1 min. and add 9 *N* H_2SO_4 (5 ml) rapidly, with vigorous swirling. Replace in the bath at 28° and, after 15 min., read the extinction at 545 mμ against a reagent blank. Recoveries in 13 replicate determinations ranged from 96 to 102%.

A. R. ROGERS

1159. **Quantitative determination of capsaicin in capsicum fruits.** Z. Kowalewski (Inst. of Medicinal Plants, Poznan). *Acta Polon. Pharm.*, 1959, **16** (4), 309–315.—After a critical examination of established methods, the following chromatographic procedure is recommended. An alcoholic extract of 0·1 g of the crude drug is transferred to a strip of paper (S. & S. 2315) and the chromatogram is developed by the descending technique with methanol - acetic acid - H_2O (5:1:5). The dried chromatogram is cut into two longitudinal strips, one of which is treated with 1 *N* NaOH and a 1% soln. of diazosulphanilic acid in 0·5 *N* H_2SO_4; in about 5 min. the capsaicin spot (R_T = 0·78) assumes a brick-red colour. The corresponding part of the other strip is cut out and eluted with 5 ml of ethanol (95%); the eluate is made alkaline with 0·1 ml of 0·2 *N* NaOH and treated dropwise with 1 ml of a 0·092% soln. of diazobenzenesulphonic acid in 0·25 *N* H_2SO_4. In acid medium the eluate assumes an orange colour; it is then treated with 1 ml of 0·075% NaI soln., warmed for 5 min. at 60° to 70° and after cooling made up to 10 ml with 0·2 *N* NaOH. The extinction is measured in a Pulfrich photometer (S50 filter) against a reagent blank. The standard curve must be prepared with cryst. capsaicin; vanillin is unsuitable as a standard. The method enables as little as 5 μg of capsaicin to be determined.

W. B. MIASKOWSKI

1160. **Tennecetin: a new anti-fungal antibiotic. General characteristics.** J. Burns and D. F. Holtman (Tennessee Univ., Knoxville, U.S.A.). *Antibiot. & Chemother.*, 1959, **9** (7), 398–405.—Details are given of the physical and chemical properties of tennecetin, including u.v. and i.r. absorption curves. Paper chromatography with water-saturated *n*-butanol and a paper disc - agar diffusion assay with *Saccharomyces carlsbergensis* are also described.

W. H. C. SHAW

1161. **Analysis of barbiturate salts in various dosage forms by an ion-exchange and non-aqueous titration procedure.** M. C. Vincent and M. I. Blake (Sch. of Pharm., N. Dakota Agric. Coll., Fargo). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (6), 359–360.—Apply a soln. of the barbiturate ($\simeq$100 mg) in dimethylformamide (20 to 25 ml) to a column of Amberlite IRC-50 (H^+ form), elute with dimethylformamide and titrate the first 50 ml of eluate with 0·1 *N* Na methoxide in benzene - methanol, with azo violet (C.I. Direct Violet 32) as indicator. The method is applicable to pure salts, ampoules, capsules, suppositories and tablets, but not to elixirs. The error is usually $< \pm 1\%$, but poor results are obtained with enteric-coated tablets of pentobarbitone calcium.

A. R. ROGERS

1162. **Determination of acetophenetidin [phenacetin] in tablet mixtures: a potentiometric method.** J. G. Baldinus and I. Rothberg (Smith Kline and French Lab., Philadelphia, Pa.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (6), 318–319.—Heat the powdered sample (containing $\simeq$170 mg of phenacetin) under reflux with 10% HCl (50 ml) for 1 to 1·5 hr., dilute with 10% HCl (50 ml), add KBr (5 g) and titrate potentiometrically with $NaNO_2$ soln., with platinum and calomel electrodes. Record the potential exactly 1 min. after each addition of $NaNO_2$ soln. Perform a blank titration on an unhydrolysed sample. The results are quant. and are not affected by the presence of caffeine, acetylsalicylic acid, salicylic acid, codeine, amphetamine, amylobarbitone and common excipients, *e.g.*, starch, talc, magnesium stearate, calcium sulphate or guar gum.

A. R. ROGERS

1163. **Oscillopolarographic behaviour of some basic substituted esters and amides (local anaesthetics).** J. Prokeš and F. Vorel (Lab. of Toxicol. and Forensic Chem., Charles' Univ., Prague). *Čas. Lék. Čes.*, 1958, **97** (6–7), 226–228.—Various natural and synthetic local anaesthetics (amylocaine, amydricaine, procaine, Intercaine, Larocaine, cocaine, pseudococaine, tropacocaine, benzamine and piperocaine) which are not polarographically reducible in *N* NaOH, produce characteristic incisions on the oscillopolarographic curve which are suitable for their detection and semi-quant. determination. The depth of the incision corresponds linearly to the concn. in the range 10^{-4} to 10^{-5} *M*. By the use of a calibration curve an accuracy of ±10% can be achieved. The method is suitable for the rapid toxicological detection of the compounds named. (*Cf.* Habersberger and Zýka, *Československ. Farm.*, 1956, **5**, 264.)

J. ZÝKA

1164. **Methods of analysis for the control of the manufacture of sulphathiazole.** A. Miss and S. Iancu. *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **7** (1), 125–136.—The normal methods, such as titration with NaOH or $NaNO_2$, cannot be used for the determination of sulphathiazole (**I**) during the manufacturing process owing to the presence of chlorides, sulphanilic acid and other impurities. Titration with Ag acetate soln. (prep. described) at pH 8 to 8·5 can be used in the presence of sulphanilic acid and chlorides, but not if the molar ratio of NaCl to **I** is >3. To determine **I** in the crude product, extraction with anhyd. acetone in a

Soxhlet apparatus is recommended, since neither NaCl nor sulphanilic acid is soluble. The extract is then buffered with ammonium acetate and titrated at pH 6 to 8·5 with Ag acetate. The acetone extract can also be titrated potentiometrically with NaOH and an antimony electrode. Titration with $NaNO_2$ often gives high results owing to the solubility in acetone of amino impurities. The acetone used for the extraction must be free from aldehydes and stored over anhydrous Na_2SO_4. H. SHER

1165. **Ionophoretic separation and determination of mixtures of salicylic acid and 2-phenylquinoline-4-carboxylic acid [cinchophen].** G. Bellomonte and A. Davidova (Ist. Sup. San., Roma, Italy). *Chim. e Ind.*, 1959, **41** (4), 308–309.—*Procedure*—The electrophoresis is carried out on strips of Whatman No. 1 paper (6 cm × 28 cm), impregnated with phosphate buffer soln. at pH 7·2, at 150 V for 3·5 hr. The soln. of the acids (as sodium salts) (0·025 ml ≡ 25 μg) is applied to the paper near the cathode. After electrophoresis, the paper is dried at 100° and the acids are revealed by their fluorescence under a Wood's lamp. The spots are cut out and eluted with H_2O, and the soln. obtained are read spectrophotometrically. Salicylic acid has an absorption max. at 296 mμ with $E^{1\%}_{1\,cm} = 272$, and cinchophen has a max. at 325 mμ with $E^{1\%}_{1\,cm} = 360$. L. ZANONI

1166. **Refractometric determination of Cardiazol [leptazol] in pharmaceutical preparations.** K. Kalinowski and I. Wardecka (Dept. of Pharm. Chem., Med. Acad., Łódź). *Acta Polon. Pharm.*, 1959, **16** (4), 325–330.—A comprehensive account is given of the refractometric method of determining leptazol in injections and tablets and in mixtures with quinine hydrochloride and ephedrine hydrochloride. Ethanol (95%) was used as a solvent for leptazol in tablets and in mixtures with quinine hydrochloride, and $CHCl_3$ for mixtures with ephedrine hydrochloride. The deviations from the results obtained by a gravimetric method did not exceed 2%. W. B. MIASKOWSKI

1167. **Evaluation of pharmaceutical organic bases with the use of ion exchangers. II.** B. Grefsgård (Ski Apotek, Norway). *Medd. Norsk Farm. Selsk.*, 1959, **21** (3), 25–31.—The method (*Ibid.*, 1956, **18**, 157) is applicable, with the use of columns of Amberlite IRA-400, to the determination of the bases in Transergan, piperidolate, Mezapine, and pipadrol. The carboxylic ester groups in the first two compounds are not attacked by the resin. The phenolic amines, pholedrine and Suprifen are eluted with difficulty from Amberlite IRA-400, but can readily be eluted from the less basic resin Amberlite IR-45. Loss of Cl^- or SO_4^{2-} from this resin can be avoided by eluting with 70% ethanol at 40° to 45°. The eluted bases are preferably titrated potentiometrically. P. S. ARUP

1168. **Microchemical differentiation of some diethylamides used in pharmacy.** G. Cavicchi and Sandri (Ist. Chim. Farm. Toss., Univ. of Ferrara, Italy). *Farmaco, Ed. Pract.*, 1959, **14** (4), 241–250.—The following compounds have been studied—nicotinic diethylamide (nikethamide), NNN'N'-tetraethylphthalamide, and the diethylamide of 3:5-dimethyl-4-isoxazolecarboxylic acid (Cycliton). Characteristic crystals are obtained with $Ni(SCN)_2$, $Mn(SCN)_2$, $Co(SCN)_2$, $HBiBr_4$ and Reinecke's salt, which serve to differentiate the compounds named. L. ZANONI

1169. **16α-Hydroxysteroids. I. Characterisation of triamcinolone.** L. L. Smith and M. Halwer (Chem. Process Improvement Dept., Lederle Lab. Div., American Cyanamid Co., Pearl River, N.Y.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1959, **48** (6), 348–352.—Triamcinolone (9α-fluoro-11β:16α:17α:21-tetrahydroxypregna-1:4-diene-3:20-dione) and its 16α:21-diacetate may be identified by determinations of m.p., [α], and u.v. and i.r. absorption spectra. The following methods are recommended for the assay of tablets. *Procedures*—Shake the powdered sample (containing ≃20 mg of steroid) with ethanol (80 ml) for 1 hr., dilute to 100 ml and centrifuge. For the polarographic determination, evaporate a 10-ml aliquot of the supernatant liquid to dryness in a stream of air and dissolve the residue in a 0·1 *M* soln. of tetra-*n*-butylammonium hydroxide in 50% aq. methanol, which has been adjusted to pH 3·0 with aq. H_3PO_4; the $E_{\frac{1}{2}}$ for triamcinolone is −1·02 V. For the absorptiometric determination, dilute an aliquot of the supernatant liquid to contain about 40 μg of steroid per ml, mix a 1-ml portion with a 0·5% soln. of blue tetrazolium in ethanol (1 ml) and a 1% soln. of tetraethylammonium hydroxide in ethanol (1 ml), set aside for 30 min. and measure the extinction at 510 to 530 mμ. A. R. ROGERS

See also Abstracts—**1116**, Morphine and other bases in urine. **1117**, Glutethimide in blood. **1181**, Thiamine in pharmaceutical preparations. **1232**, Water in tobacco.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1170. **High-frequency titration of halogens in food.** Ihei Iwata (Dept. of Agric. Chem., Fac. of Agric., Univ., Hongo, Tokyo). *Japan Analyst*, 1959, **8** (3), 149–152.—By the use of a condenser-type high-frequency apparatus (quartz oscillator, 455 kilocycles) equipped with a cathode ray tube, a point of inflection is clearly observed in pptn. titrations with 0·001 *N* $AgNO_3$. The deviation is ±0·3 ml. Large amounts of electrolytes interfere. The sample (0·5 g) is ignited with Na_2CO_3 (2·5% by wt. of the sample) and distilled in a current of O, in the presence of $K_2Cr_2O_7$, $Ag_2Cr_2O_7$ and H_2SO_4. The halogens are absorbed in NaOH soln. containing H_2O_2, acidified with HNO_3, and mixed with a known excess of 0·001 *N* $AgNO_3$, and the excess is back-titrated with 0·001 *N* NH_4SCN. K. SAITO

1171. **Determination of DDT residues in foodstuffs.** G. A. Sergeant and R. Wood (Dept. of the Gov. Chemist, Clement's Inn Passage, Strand, London). *Analyst*, 1959, **84**, 423–426.—Fatty material is dissolved in hexane or the fat content is extracted with hexane, the soln. is treated gradually with 40 ml of H_2SO_4, then set aside for 30 min. The hexane layer is applied to a column of acidified Celite and the percolate is reduced to a vol. of a few ml. The residue is washed with hexane on to a silica gel column, which is then washed with hexane and drained under pressure. The DDT is eluted with ether, the eluate is evaporated to low bulk and finally to dryness at 40° by means of a current of dry air. After 30 min. on a steam bath the residue is nitrated with a mixture of H_2SO_4 and HNO_3, the derivative is extracted with a light petroleum - benzene mixture, and the extract is washed with alkali and evaporated to dryness at

low temp. The residue is dissolved in benzene, ethanolic KOH is added, and the extinction is measured at 600 mμ and referred to a calibration graph prepared from *pp'*-DDT by the same method of nitration. Determinations of known amounts in a number of fatty food materials, which included butter, milk and olive oil, gave recoveries ranging from 86 to 95%. A. O. JONES

1172. **Improved method for the estimation of uric acid in insect-infested foodstuffs.** S. V. Rao, K. Krishnamurthy, M. Swaminathan and V. Subrahmanyan (Central Food Technol. Res. Inst., Mysore). *Food Sci., Mysore*, 1959, **8** (4), 121–122.—The reagent of Benedict and Franke (*J. Biol. Chem.*, 1922, **52**, 387) is known to indicate the presence of uric acid (**I**) in uninfested foodstuffs when none can be detected by paper chromatography. This is due to interfering materials that give an "apparent" value for **I**. The "total" **I** in a sample of infested foodstuffs is determined by the method of Benedict and Franke. A similar sample is treated first with uricase at pH 9·2 to destroy **I** and then with Benedict's reagent to obtain the "apparent" content of **I**. The true amount of **I** is obtained by difference. Infested black gram (*Phaseolus mungo*) contained considerably larger quantities of **I** than did infested wheat flour. S. G. AYERST

1173. **Malt bread and "pasta." [Detection of the addition of malt.]** F. Di Stefano and A. Vercillo (Ist. Sup. Sanità, Roma, Italy). *Boll. Lab. Chim. Provinciali*, 1959, **10** (2), 111–115.—*Procedure*—In a 200-ml flask place 10 g of dried and ground bread, add 100 to 150 ml of H_2O and set aside at 40° for 30 min.; add 5 ml of 15% $K_4Fe(CN)_6$ soln. and 5 ml of 23% Zn acetate soln., shake, cool and make the soln. up to the mark. Filter, and determine the reducing sugars as maltose. L. ZANONI

1174. **Determination of trimethylamine oxide in fish muscle.** J. Bystedt, L. Swenne and H. W. Aas (Svensk. Inst. f. Konserveringsforsking, Göteborg, Sweden). *J. Sci. Food Agric.*, 1959, **10** (6), 301–304.—Of a number of reagents for reducing trimethylamine oxide to trimethylamine (**I**), the best is $TiCl_3$. *Procedure*—To 20 ml of an extract of fish muscle in 5% trichloroacetic acid add 0·5 ml of 10% $TiCl_3$ soln. and set aside for 2 hr. The excess of $TiCl_3$ is then removed by the addition of a few drops of satd. $KMnO_4$ soln. After 5 to 10 min. dilute to 100 ml with 5% trichloroacetic acid soln. and determine **I** colorimetrically as the picrate (Dyer, *Brit. Abstr. C*, 1946, 110), with a calibration curve prepared with trichloroacetic acid soln. of **I**. The coeff. of variation due to sampling and analytical error are discussed. P. D. PARR-RICHARD

1175. **Simplified determination of ash in the general analysis of cheese.** E. Pijanowski. *Roczn. Technol. i Chem. Żywności*, 1959, **4**, 47–57.—The sample (2 to 4 g) is incinerated to a white ash in a platinum or porcelain dish. Alkali-metal salts need not be removed. The ash is weighed, and the Cl^- therein are determined by Volhard's method. It is assumed that the loss of Cl^- during ignition is due partly to the influence of the P (0·71%) in the casein (1 mole of P causing the loss of 2 moles of Cl^-) and partly to volatilisation of NaCl. The true ash content (A) is given by the formula—

$$A\ (\%) = [(100a - 0{\cdot}585n)/m] - (k/70 + S),$$

where a is the observed wt. of ash (g), n is the vol. (ml) of 0·1 N $AgNO_3$ used in the titration of the Cl^-, m is the wt. of sample (g), and k is the casein (%) and S the NaCl (%) in the sample; k can be calculated from the equation $k = 100 - (w + f + 6{\cdot}5)$, where w is the H_2O (%) and f is the fat (%) in the sample. The method gives results accurate to within 1%, and is two or three times as rapid as the standard procedure. Too rapid an incineration (<3 hr.) and the use of NH_4NO_3 as an incineration aid led to a considerably reduced accuracy. A. GROCHOWSKI

1176. **[Determination of] non-volatile organic acids in fresh unripe pickling mangoes and salted mango slices by paper chromatography.** N. L. Jain, G. V. Krishnamurthy and G. Lal (Central Food Technol. Res. Inst., Mysore). *Food Sci., Mysore*, 1959, **8** (4), 115–117.—Concentrated extracts of fresh mango slices, and of salted slices that had been stored for a year, were examined by descending paper chromatography, with *n*-butanol - formic acid - water (12:5:15) as solvent and bromocresol green as spray reagent. Oxalic, citric, malic, succinic and ascorbic acids and two unknown acids were present in fresh mangoes, and all these acids, except ascorbic, in the salted slices. S. G. AYERST

1177. **Isolation of resins from vanilla by chloroform - amine extraction.** H. P. Burchfield and E. A. Prill (Boyce Thompson Inst. for Plant Res., Yonkers, N.Y.). *Contr. Boyce Thompson Inst.*, 1959, **20** (2), 135–139.—Resins are removed from single-strength vanilla extract containing 35% of ethanol by a first extraction with $CHCl_3$ containing 3% of 1:3-di-*o*-tolylguanidine (**I**), and second and third extractions with the same solvent containing 8% of **I** and 20% of ethanol. After evaporation of the $CHCl_3$, the resins are liberated from their guanidinium salts by treatment with acetic acid and HCl, and are then recovered by filtration, and separated into tetrahydrofuran-soluble and methanol-soluble fractions for determination by weighing. Possible methods of applying the procedure to the isolation of other components of plant extracts are suggested. M. D. ANDERSON

1178. **Rapid method for the determination of benzoic acid in soft drinks.** E. English (Counties Public Health Lab., 66, Victoria St., London). *Analyst*, 1959, **84**, 465–466.—The sample is diluted to give an expected concn. of $\simeq$4 p.p.m. of benzoic acid and is filtered, with rejection of the first 50 ml of filtrate. The extinction (x) of the subsequent portion is measured at 230 mμ. A blank measurement (y) is made with a prepared sample of the drink containing a known concn. (a) (p.p.m.) of benzoic acid. The concn. in the sample is then ax/y. The use of the reference standard prepared as described is essential because the unpreserved product, as well as having a characteristic extinction of its own at 230 mμ, has an influence on the extinction of benzoic acid. A. O. JONES

1179. **Determination of diastatic power in malt extracts.** F. Muntoni (Ist. Sup. Sanità, Roma, Italy). *Boll. Lab. Chim. Provinciali*, 1958, **9** (4), 405–413.—*Reagents*—(*i*) Phosphate buffer is prepared by mixing 95 ml of 0·067 M KH_2PO_4 with 5 ml of 0·067 M $Na_2HPO_4.2H_2O$. (*ii*) Starch (7·5 g) is ground with 25 ml of phosphate buffer soln., diluted with 220 ml of water, and heated in a 300-ml flask, first at 45° and then by immersion

in a boiling-water bath, with shaking until the starch dissolves. After 30 min. the soln. is cooled to 40°. *Determination of pre-existing sugars*—Malt extract (1 g) is dissolved in water and transferred to a 100-ml flask, 5 ml of basic Pb acetate soln. 10% (w/v) is added followed after 10 min. by 10 ml of satd. Na_2SO_4 soln.; after a further 10 min. the contents are made up to vol. and reducing sugars are determined as maltose (*Z*). *Determination of saccharifying power*—To the 300-ml flask containing the starch soln. at 40° is added 10 ml of malt extract soln. (2 g of malt extract dissolved in 10 ml of buffer soln. and made up to 100 ml with water) and the mixture is maintained at 40° with frequent stirring for 30 min. It is then neutralised with 3 ml of KOH soln. (10%, w/v), cooled and made up to vol. The reducing sugars are expressed as vol. (ml) of soln. (*R*) required to reduce 10 ml of Fehling's reagent (diluted 1:4). The diastatic power, expressed in Pollack units, is given by $111150/R - 10Z$.
L. Zanoni

1180. **Paper chromatography of volatile fatty acids.** Himansu Mukerjee (Univ. Coll. of Sci. and Technol., Calcutta, India). *Anal. Chem.*, 1959, **31** (7), 1284.—The separation of 2 to 5 μmoles of the volatile fatty acids produced during the fermentation of certain algae (*Chlorella vulgaris* and *Chlorella pyrenoidosa*) is achieved by descending paper chromatography of their ammonium salts at 26° ± 1°, with methanol - acetone - 30% aq. NH_3 (70:30:1). Methanol containing 1 ml of 30% aq. NH_3 per 100 ml satisfactorily separates ammonium formate and acetate.
J. P. Stern

1181. **Microbiological methods of determination of thiamine.** K. Myszkowska, J. Tautt, S. Tuszyńska and W. Woźniak (Drug Res. Inst., Warsaw). *Acta Polon. Pharm.*, 1959, **16** (4), 317–324.—Microbiological methods for the determination of thiamine in pharmaceutical preparations, body fluids, yeasts and milk have been studied, with *Lactobacillus fermenti* and *Phycomyces blakesleeanus* as test organisms. The procedures are described and results quoted, and suggestions are made regarding the suitability of the individual methods for the analysis of various test samples.
W. B. Miaskowski

1182. **Comparison of two species of lactobacilli as test organisms in pantothenic acid estimation. *Lactobacillus casei* and *Lactobacillus plantarum*.** M. F. Clarke (Dept. of Physiol. Chem., Woman's Med. Coll., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1243–1246.—*Lactobacillus casei* (**I**) and *Lb. plantarum* (**II**) are compared on an enriched medium containing enzyme-hydrolysed casein and 3% of glucose. With **II** the acid produced is directly proportional to the log of the Ca pantothenate added for doses from 0·03125 to 0·5 μg. For **I** the slope is steeper and the proportionality is limited to the log of doses from 0·03125 to 0·125 μg. Blanks with **I** are also higher. *Procedure*—Make stab transfers of **I** and **II** to the medium and incubate at 37°. After 20 to 24 hr. centrifuge the culture, wash with 0·85% saline, and re-suspend in saline to the original vol. Dilute to a reading of 90 to 100 in a Klett - Summerson colorimeter fitted with a red filter. Add one drop of this inoculum to 10 ml of soln. containing Ca pantothenate and incubate at 37° for 70 to 72 hr. Determine the acid produced volumetrically, with phenolphthalein as indicator.
J. P. Stern

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1183. **Indicator tube for the determination of trichloroethylene in air.** J. C. Gage (I.C.I. Ltd., Ind. Hygiene Res. Lab., Welwyn, Herts., England). *Analyst*, 1959, **84**, 509–515.—The air sample is drawn through two tubes in series, the first charged with silica gel impregnated with $KMnO_4$ and H_3PO_4 and the second with silica gel impregnated with *o*-tolidine hydrochloride. The second tube measures the Cl liberated in the first and is calibrated to relate the concn. of Cl to the length of stain produced. The method is suitable for routine industrial hygiene analysis of air containing trichloroethylene vapour.
A. O. Jones

1184. **Automatic atmospheric fluoride pollutant analyser.** D. F. Adams and R. K. Koppe (Div. of Ind. Res., State Coll. of Washington, Pullman, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1249–1254.—The instrument described produces a continuous integrated record of fluoride concn. for finite periods and consists essentially of a circulating fluoride-sensitive reagent, which is continuously brought into contact with freshly sampled air, and a recording flow colorimeter. Fresh reagent is fed into the system on a pre-set time schedule, which is automatically modified if the accumulated fluoride concn. exceeds a pre-determined level. Good correlation has been established with conventional sampling procedures.
K. A. Proctor

1185. **Determination of heavy water in concentrations approximating to that in natural water.** I. J. L. Otero de la Gandara and M. Gispert Benach. *An. Real Soc. Esp. Fis. Quim., Madrid, B*, 1959, **55**, 73–82.—A simple inexpensive apparatus is described which can be used to determine D_2O in natural water by the float method. The D_2O in natural water is concentrated to a convenient degree by electrolysis in four electrolytic cells. Two controls and two unknowns are run simultaneously in series. The method gives results with a max. relative error of 3%.
Nucl. Sci. Abstr.

1186. **Spectrographic analysis of water for chlorine, bromine, iodine and sulphur.** B. Ya. Kudȳmov, V. I. Malinina and V. P. Varlamov (All-Union Sci. Res. Geological Prospecting Petroleum Inst.). *Zavod. Lab.*, 1959, **25** (5), 583–584.—The soln. in a fulgurator is sparked between carbon electrodes, and intensities of lines in the visible region are used for determining Cl, Br, I and S.
G. S. Smith

1187. **Determination of bromine in natural waters.** L. N. Podgornȳĭ and F. I. Bezler. *Byul. Inst. Biol. Vodokhranilishch. Akad. Nauk SSSR*, 1959, (2), 56–58; *Ref. Zhur., Khim.*, 1959, (14), Abstr. No. 49,272.—A modified colorimetric method with fuchsine (**I**) is described. The sensitivity of the method is greatest at a concn. of < 3 mg of Br^- per ml and the colour stability is increased if H_2O_2 is used as the oxidising agent instead of $K_2Cr_2O_7$. Ammonium molybdate soln. is added to the soln. at the same time as the H_2O_2 to inhibit the oxidation by the latter of the bromine derivative of **I**. Standards are prepared from KBr soln. (0·01 mg per ml) (1 to 9 ml diluted to 10 ml); 1% ammonium molybdate soln. (2 drops), conc. HCl (8 drops), conc. H_2O_2 (7 drops) and conc. H_2SO_4 (2 ml) are added, the mixture is cooled to room temp., 1 ml of a soln. of **I** is added [dissolve basic fuchsine (1 g) in H_2O (1 litre) and add 100 ml of this soln. to H_2SO_4

(1:20) (1 litre)], and the soln. is extracted with $CHCl_3$ (2 ml); the colour takes 10 to 15 min. to develop. The test soln. is treated in the same way.
K. R. C.

See also Abstracts—**864**, Metals in mineral waters. **886**, Ni and Cu in natural waters. **901**, Zn in natural waters. **956**, H_2S and CO_2 in air. **1188**, CO_2 and O in soil air. **1191**, Water bath for B.O.D. determinations.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1188. **Determination of carbon dioxide and oxygen in soil air.** B. N. Makarov. *Pochvovedenie*, 1959, (1), 121–125; *Ref. Zhur., Khim.*, 1959, (13), Abstr. No. 45,604.—In the simple method proposed, the sampling of the soil air and the determination of the concn. of CO_2 and O are carried out directly in the field in the same flasks, fitted with ground-glass stoppers; CO_2 is absorbed in $Ba(OH)_2$ soln.; O is determined by the oxidation in alkaline medium of Mn^{2+} to Mn^{3+} and Mn^{4+}, which in acid medium oxidise I^- to iodine, and the iodine is titrated with thiosulphate.
C. D. KOPKIN

1189. **Macro- and micro-gravimetric methods for the determination of mercury in plant-protective substances.** D. Pirtea and I. Albescu (Centrul de Cercet. Chim., Acad. R.P.R., București, Romania). *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **7** (1), 137–147.—The proposed method is a combination of the method of Walton and Smith (*Anal. Chem.*, 1956, **28**, 406) for the oxidation of organic matter, and a modification of the method of Spacu and Suciu (*Z. anal. Chem.*, 1929, **78**, 244) for the determination of Hg. Iron, Al, Zn, Mg, Mn, Ni and Co are masked with EDTA (disodium salt). *Procedure for macro-determination*—The sample (0·5 to 3·5 g) is boiled with a mixture of 12 ml of an aq. soln. containing 6 g of KI and 0·6 g of iodine and 3 ml of conc. H_2SO_4, the time depending on the insecticide present. Phenylmercury acetate (**I**) requires 5 min., phenylmercury chloride (**II**) 15 min.; a mixture of **I** or mercuric acetate with **II** also requires 15 min.; methoxyethylmercury silicate requires 30 min. After boiling the mixture it is diluted to 50 ml with boiling water and filtered. The residue is washed with 50 to 150 ml of boiling water. (The more Fe, Al, Zn, Ni and Mn present, the greater is the dilution required.) The excess of iodine is removed by the dropwise addition of Na_2SO_3 soln. (10%) until the colour changes from brown to yellow. An excess of Na_2SO_3 will lead to the formation of cuprous mercuric iodide when the reagent is added; to avoid this, a little KI can be added if too much Na_2SO_3 has been added. Enough EDTA (disodium salt) (0·5 to 1·0 g) is added to complex the contaminating metals, followed by the dropwise addition of conc. aq. NH_3 to pH $\simeq$7. The soln. is boiled and treated with a boiling satd. soln. of the reagent $(Cuen_2)(NO_3)_2.2H_2O$ (**III**) (en = ethylenediamine) until the soln. becomes a deep violet (an excess is required, since an excess of EDTA decomposes the reagent). The mixture is cooled in ice, stirred and set aside for 30 min. for the pptn. of pale mauve crystals of $[HgI_4][Cuen_2]$. The ppt. is washed 5 to 6 times on a sintered crucible with KI soln. (0·1%) and **III** (0·1%), 2 to 4 times with ethanol and diethyl ether, and dried *in vacuo*. *Procedure for micro-determination*—The sample (0·01 to 0·05 g) is treated with 0·5 to 2 ml of the oxidising reagent as for the macro-method, the vol. of the final soln. being $\geq$10 ml. This is treated with Na_2SO_3, EDTA (disodium salt) (0·05 g) and conc. aq. NH_3, and **III** is added in the cold. The ppt. is washed on a filter-stick 4 to 6 times with 1 to 2 ml of the KI reagent soln. as described above, then with 96% ethanol, and then with ether. With grain, it is recommended to extract the insecticide first with 2-ethoxyethanol or with methanol.
H. SHER

See also Abstracts—**962**, Determination of SO_4^{2-} in superphosphate. **1122**, Carbohydrates of grasses. **1123**, Analysis of grasses. **1126**, Extraction of plant pigments. **1171**, DDT residues in food.

5.—GENERAL TECHNIQUE AND APPARATUS

General

1190. **Wash-bottle for dispensing carbon dioxide free water.** R. A. Latimer (Science Serv. Lab., Dept. of Agric., London, Ontario, Canada). *Chemist Analyst*, 1959, **48** (2), 46.—A one-way polyethylene valve is inserted into a hole drilled in the shoulder of a one-pint polyethylene bottle with the outlet inside the bottle. An absorption tube containing a NaOH - asbestos absorbent mixture is attached to remove CO_2. Other substances can be removed by use of appropriate absorbents. A second one-way valve is inserted through the bottle-cap opening, and is fitted with an extension tube to the bottom of the vessel and a delivery tip drawn and bent to the required shape.
S. M. MARSH

1191. **Water bath for the determination of the biochemical oxygen consumption in 5 days at 20° in water and effluent water.** R. Schutte and H. J. Schaafsma (Lab. Tech. Adviesbureau der Vereniging van Nederlandse Gemeenten, Tesselschadelaan 4, Amersfoort, Netherlands). *Chem. Weekbl.*, 1959, **55** (44), 612–613.—The construction of a water bath is described in detail. The insulation is such that, when the bath is kept at 20°, the pump and the heating element are seldom used, even when the apparatus is opened frequently. The water level in the bath is kept constant and does not change when a large number of bottles is placed in the bath or taken from it. Even at high summer temperatures only a little cooling was necessary.
M. J. MAURICE

1192. **Modified glass still for fluoride analysis by pyrohydrolytic separation.** P. B. Adams and J. P. Williams (Corning Glass Works, Corning, N.Y., U.S.A.). *Chemist Analyst*, 1959, **48** (2), 48.—The apparatus described previously for the determination of fluoride in glass and silicate materials by pyrohydrolysis at 1000° (*J. Amer. Ceram. Soc.*, 1958, **41**, 377) has been modified to give a more convenient and compact unit. A detailed illustration is given.
S. M. MARSH

1193. **Design of Teflon vessel for decomposition of rock samples.** L. Shapiro (U.S. Geological Survey, Washington, D.C.). *Chemist Analyst*, 1959, **48** (2), 46.—A simply-made Teflon vessel is described which has a close-fitting lid to prevent the evaporation of HF during digestion, and which when

placed in a hole in sheet aluminium or hardened plastic can be immersed in steam; 30 or more samples have been successfully treated overnight in such vessels, the process being completed by heating for about 1 hr. to evaporate HF. Excessive local heating must be avoided and the last traces of fluoride should be removed after transferring the sample with a minimum of water to a 400-ml Vycor or pure silica beaker. The advantages of this transfer are that traces of fluoride combine with the glass of the beaker and are evaporated, and the capacity of the beaker permits swirling and the addition of HNO_3 to destroy organic matter and also the addition of sufficient water to dissolve soluble sulphates. S. M. MARSH

1194. **Pipette with a removable reservoir for use in micro-deproteinisations.** M. Herbain (Serv. Rech. Roussel-Uclaf, Romainville, Seine, France). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 633–636.—The pipette (illustrated), which is in two parts assembled by means of a ground glass joint, consists of a calibrated capillary tube and a graduated reservoir, this serving to give an accurate measure of the final volume of deproteinised soln. The whole assembly is used in the same way as a haematological pipette, the liquid to be deproteinised, the deproteinising fluid and the rinsing soln. being measured by the pipette directly into the reservoir whilst it is still attached to the pipette. After mixing the reactants, assisted by the addition of a few small glass spheres, the reservoir is stoppered and inverted. The pipette is removed, the outlet of the reservoir is stoppered, and the reservoir and its contents are centrifuged. The recovery of the deproteinised liquid is almost complete. The volumes of the pipette and reservoir can be modified as desired. D. B. PALMER

1195. **Automatic stopcock turner.** D. N. Hume and J. L. Paige (M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1286.—Instead of the elaborate solenoid system of Malmstadt for the automatic or remote operation of stopcocks (*Ibid.*, 1957, **29**, 1901), a device that can be assembled and started within minutes is described. The actuating element is a small electric motor, and the torque developed will turn burette or vacuum stopcocks. W. J. BAKER

1196. **Turbidimetric titrations.** R. B. Fischer, M. L. Yates and M. M. Batts (Indiana Univ., Bloomington, U.S.A.). *Anal. Chim. Acta*, 1959, **20** (6), 501–505.—The addition of the non-ionic detergents Tergitol and Triton improves the conditions for certain turbidimetric titrations, *i.e.*, titrations to an end-point of max. turbidity. Their effect is to induce further particle formation as opposed to the growth of particles already formed. Titrations favourably affected are those that involve the pptn. of $BaSO_4$, $PbCO_3$, Ca oxalate, Ni dimethylglyoxime and CaF_2. There is no significant effect on the pptn. of $PbMoO_4$ and AgCl. Dextrin enhances the nucleation of $PbMoO_4$. H. N. S.

Chromatography, ion exchange, electrophoresis

1197. **Simple apparatus for back-washing chromatographic columns with inert solvents.** R. L. Dannley and B. L. Weigand (W. Reserve Univ., Cleveland, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1284–1285.—The apparatus described enables the solvent to be re-used; this is drawn from a reservoir, filtered through a glass-wool plug, forced by a pump through the column, and then returned to the reservoir. Fines from the column are removed in the reservoir by passage through a glass-wool filter held in place by glass beads.
J. P. STERN

1198. **Drop-counting fraction-collector accessories.** A. C. Arcus (Nutrition Res. Dept., Med. Sch., Dunedin, New Zealand). *Anal. Chem.*, 1959, **31** (7), 1282.—Modifications to a drop-counting fraction collector to adapt it for use with gradient elution chromatography are described.
K. A. PROCTOR

1199. **Purification of formamide for chromatographic use.** J. R. Cole, J. O. Knox and A. L. Picchioni (Coll. of Pharm., Univ. of Arizona, Tucson, U.S.A.). *Chemist Analyst*, 1959, **48** (2), 38.—A 20% suspension of charcoal in commercial formamide was shaken at regular intervals for 4 hr. The charcoal was then filtered off. The purified formamide was satisfactory for use in the paper chromatography of steroids. It had a sp. gr. of 1·1331 and a refractive index of 1·4428, both at 25°.
S. M. MARSH

1200. **Chromatography by vapour entrainment.** C. Dumazert and C. Ghiglione (Lab. de Chim., Fac. de Méd. et de Pharm., Boulevard d'Alès, Marseille, France). *Bull. Soc. Chim. France*, 1959, (4), 615–617.—An apparatus is described in which a glass U-tube, 3 metres long and 8 to 10 mm in diam., is covered with asbestos cloth threaded with resistance wire as a heater; the end of one arm is fitted with a thermometer and leads to a micro-condenser, and the end of the other arm is closed by a stopper, but communicates, through small holes, with a jacket surrounding it for the first 50 cm. The lower end of this jacket communicates with an evaporating space provided with a heater into which the volatile solvent used as mobile phase is dripped from a burette under a constant head. For the separation of fatty acids the U-tube is charged with acid-washed sieved refractory brick, impregnated with liquid paraffin (15 g) and stearic acid (1·5 g). Ethanol vapour is used as the mobile phase at a temp. of 95° to 125°. The sample (4 to 5 μg) in ethanol (10 to 20 μl) is added rapidly under an ethanol atmosphere to Gooch asbestos in a small open-ended tube at the entry end of the U-tube, and the stopper is rapidly replaced. The distillate from the exit end is collected at intervals of 2 to 5 min. and titrated with 0·005 *N* NaOH. For a flow rate of 20 to 60 μl of ethanol per min., retention vol. are characteristic, and vary with temp., but only slightly with flow rate. Acetic, propionic and *n*-butyric acid are completely separated. A commercial sample of *n*-valeric acid was found to contain three acids of similar b.p.
E. J. H. BIRCH

1201. **Gas chromatography. Effect of sample size on height of equivalent theoretical plate and retention volume.** R. M. Bethea and M. Smutz (Chem. Engng Dept., Iowa State College, Ames, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1211–1214.—The effect of sample size at low gas-flow rates has been studied with certain alcohol and ester samples on dibutyl phthalate and dibutyl sebacate columns. Minimum values of HETP (height equivalent to a theoretical plate) are recorded in each case. K. A. PROCTOR

1202. **Gas and liquid elution chromatography. Quantitative detector evaluation.** H. W. Johnson, jun., and F. H. Stross (Shell Devel. Co., Emeryville, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1206–1211.—Desirable detector properties are discussed and a specific evaluation procedure is proposed. A quant. treatment of noise is developed and this makes possible a precise statistical definition for the limit of detection. K. A. PROCTOR

1203. **Gas chromatograph. Ionisation by α-particles for detection of the gaseous components in the effluent from a flow reactor.** W. M. Graven (Dept. of Chem., Univ. of Oregon, Eugene, U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1197–1199.—Multicomponent gaseous samples can be analysed with a gas-adsorption chromatograph in which the ionisation current produced by α-particles from an aged Ra-D source are used to detect the components in the percolate from a column packed with molecular sieves. This type of detector is insensitive to fluctuations in temp. or gas-flow rate and is moderately sensitive to all permanent gases except H. K. A. PROCTOR

1204. **Sample-injection valve for gas chromatography.** E. P. Samsel and J. C. Aldrich (Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1288.—The valve described enables a volatile liquid to be injected into a sample chamber maintained momentarily under no pressure, while the carrier gas flows unrestricted through the chromatographic column. The valve is usually operated at $<100°$, but has been operated at 250°. It has proved satisfactory for routine use, especially in the separation of highly volatile liquids. K. A. PROCTOR

1205. **Trap for liquid fractions separated by gas chromatography.** G. J. Frisone (Rohm & Haas Co., Philadelphia, Pa., U.S.A.). *Chemist Analyst*, 1959, **48** (2), 47.—The U-tube trap is designed with a well at the bottom from which liquid fractions may be easily removed for further study. A dimensioned diagram is given. S. M. MARSH

1206. **Simple apparatus for electrophoresis.** F. Rappaport and M. Rabinovitz (Beilinson Hosp., Petah-Tiqvah, Israel). *Clin. Chim. Acta*, 1959, **4** (4), 531–534.—The apparatus operates on a low voltage (50 to 60 V) and is inexpensive and can be assembled from readily available materials without technical skill. Its use with paper and cellulose media is described. H. F. W. KIRKPATRICK

1207. **New apparatus for zone electrophoresis on starch [columns].** R. Bourrillon and R. Got (Lab. Biochim., Fac. de Méd., 45 rue des Saint-Pères, Paris). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 643–647.—A plastic tube, which can be separated longitudinally, is used to hold the starch column. The column is prepared in two stages, the sample being introduced after the first stage, and the separation is carried out in the standard manner, with the column held vertically. When the process is complete, the starch column is removed, cut into sections, and the separated compounds are eluted. The advantages of this technique include a more regular solvent front, and no displacement during sectioning of the column. D. B. PALMER

1208. **Study of paper protein electrophoresis with special reference to a new method of sub-fractionation.** W. B. Yeoman (Frenchay Hosp., Bristol, England). *Clin. Chim. Acta*, 1959, **4** (4), 523–530.—By the method described, serum proteins are separated into 11 fractions. Barbitone buffer containing added Ca lactate (384 mg per litre) and Whatman chromatographic paper No. 1 are used. A potential of 175 V is applied for 16 hr. The strips are stained in methanolic azocarmine B, and scanned in a reflectance densitometer, allowance being made for albumin tailing. H. F. W. KIRKPATRICK

Optical

1209. **Internal standard method of general spectrographic analysis.** F. M. Smith (General Electric Co. Hanford Atomic Products Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-59864, 1959, 15 pp.—A method of moderate accuracy was developed for the analysis of diverse materials. The method was studied by the preparation of working curves for the elements Cr, Fe, Ni, Co, Al, Mo, Cu, B, Mg, Mn, Sn, Si, U, Bi, Be, Pb, Ti, Ag, Zn and Zr. These can be determined with an average deviation of $\pm 15\%$ and with an accuracy within 20%. The powdered sample is mixed with a diluent containing germanium dioxide, graphite and an internal standard, gallium oxide. A portion of this mixture is packed into a graphite electrode cup and arced at 15 amp. d.c. for 120 sec. NUCL. SCI. ABSTR.

1210. **Visual spectrophotometer for emission analysis.** Anon. *ChemikerZtg*, 1959, **83** (12), 413–414.—The Bauart Fuess spectrophotometer is described. It appears to be a development of an earlier model (*cf.* Tyas, *Anal. Abstr.*, 1957, **4**, 752). A polarising optical system presents the spectrum of the sample in a split-field eyepiece, one spectrum above the other, with a third spectrum, that of a reference sample, in register with the upper spectrum, at the bottom. The middle spectrum may be moved laterally. When the two sample spectra are in alignment, reference to the standard spectrum permits qual. analysis. By moving the middle spectrum laterally a selected line may be brought into alignment with a chosen reference line in the standard spectrum and, by rotation of a calibrated polarising lens, the intensity of the sample line may be reduced until it matches that of the reference line; quant. results may then be obtained by reference to a calibration graph. [Although not described in the text, the illustration suggests that a camera may be attached.] T. R. ANDREW

1211. **Automatic recording systems for use with a high-dispersion spectrograph.** E. W. T. Richards and M. D. Crew (Chem. Div., Res. Group, A.E.R.E., Harwell, England). A.E.R.E. Report AERE-R3035, 1959, 5 pp.—Two methods of direct recording are described. In the first, the spectrum is scanned rapidly across the entrance slit of a photomultiplier by means of a rotating mirror system, and the signal is displayed on an oscilloscope. In the second, the rotating mirror moves the spectrum slowly across the photomultiplier slit, and the signal is amplified by a suitable amplifier-integrator circuit and fed to a chart recorder. G. SKIRROW

1212. **Micro gas cell for infra-red spectroscopy.** J. U. White, N. L. Alpert, W. M. Ward and W. S. Gallaway (White Devel. Corp., Stamford, Conn.,

U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1267–1270.—The multiple-traversal cell described has a path length of 1 metre and a vol. of 22 ml. It has been used with a Beckman IR-5 spectrophotometer to observe the spectra of gas-chromatographic fractions $<0{\cdot}05\%$ of the total charge.

K. A. PROCTOR

1213. **Sampling technique for infra-red spectroscopy of solids.** J. E. Stewart (Beckman Instruments, Inc., Fullerton, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1287.—The technique described, of squeezing the sample between alkali-metal-halide windows, has proved useful in obtaining satisfactory spectra of a number of solid materials which, because of their volatility or unsuitable refractive index, are unsuitable for use with the normal pressed disc procedure. The technique can be used only with relatively soft materials. K. A. PROCTOR

1214. **Raman spectra in spectrofluorimetry.** C. A. Parker (Admiralty Materials Lab., Holton Heath, Poole, Dorset, England). *Analyst*, 1959, **84**, 446–453.—The effect of the Raman spectra of some solvents on the measurement of the fluorescence of very dil. soln. has been investigated. Solvents that contain H atoms, *e.g.*, water, ethanol and *cyclo*hexane, show a Raman band at a wave number $\simeq 0{\cdot}3\ \mu^{-1}$ lower than that of the exciting light, and this can produce appreciable distortion of the fluorescence spectrum of a strongly fluorescent substance at a concn. of $0{\cdot}1\ \mu$g per ml or less. The main Raman band of CCl_4 is so near that of the exciting light (Hg bands) that it is unlikely to interfere with fluorescence spectra. The high-frequency band of $CHCl_3$ is less intense than those of the other solvents (*supra*) and is thus likely to cause less interference. The importance of the correct choice of secondary filters in high-sensitivity fluorescence measurements is discussed.

A. O. JONES

1215. **Simple ultra-violet photometer.** R. E. Thiers, M. Margoshes and B. L. Vallee (Biophys. Res. Lab., 721, Huntington Ave., Boston, Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1258–1261.—The instrument described is small, sturdy and of simple construction and is suitable for routine work when measurements in the 355-mμ region are required. A monochromatic light beam, of band width 40 mμ, is obtained from a commercial fluorescent lamp. The detectors are photo-electric cells which operate a d.c. micro-ammeter. The precision of the instrument is about the same as that of conventional spectrophotometers.

K. A. PROCTOR

1216. **Microscope phase fluorimeter for determining the fluorescence lifetimes of fluorochromes.** B. D. Venetta (Dept. of Anatomy, W. Reserve Univ. Sch. of Med., Cleveland, Ohio, U.S.A.). *Rev. Sci. Instrum.*, 1959, **30** (6), 450–457.—Sinusoidally modulated light passes through the fluorochrome, bound to a thin polymeric substance, and is observed by a microscope in which the eye-piece is replaced by a photomultiplier tube. From the phase-shift between the unabsorbed light passing through the sample and the fluorescent light it is possible to estimate the mean fluorescence lifetime of the sample. The mean fluorescence lifetime ($2{\cdot}7 \times 10^{-9}$ sec.) of proflavine can be determined with a precision of 4×10^{-10} sec. G. SKIRROW

1217. **Automatic sulphate ion analyser.** H. N. Claudy, F. W. Karasek, B. O. Ayers and J. G. Skinner (Phillips Petroleum Co., Bartlesville, Okla., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1255–1258.—The instrument described is used for the continuous turbidimetric measurement of SO_4^{2-} in the manufacture of phosphoric acid. The SO_4^{2-} are pptd. with $BaCl_2$ in an acid gelatin soln. The resulting colloidal sol is stable, and reproducible results are obtained. K. A. PROCTOR

See also Abstract—**1184**, Automatic fluoride analyser for air.

Thermal

1218. **Gas-burner with coupled gas and air supply.** P. Spaander and H. J. Elskamp (Lab. Bodem, Water en Lucht, Rijks Inst. v. de Volksgezondheid, Utrecht, Netherlands). *Chem. Weekbl.*, 1959, **55** (20), 232–233.—A Landman burner is so modified that the gas supply and the air supply can be regulated simultaneously. Details are given. A set of four such burners has been constructed, by means of which the reproducibility of results is improved, owing to the fact that the boiling of soln. in this apparatus is more reproducible than in the commonly used ones. M. J. MAURICE

1219. **A simple method for obtaining melting-points of compounds reacting with moisture or oxygen.** A. G. Pinkus and P. G. Waldrep (Dept. of Chem., Baylor Univ., Waco, Texas). *Mikrochim. Acta*, 1959, (5), 772–774 (in English).—The solid of which the melting-point is to be determined is powdered in a dry-box in an atmosphere of dry nitrogen, and is introduced into the capillary inside the box. D. F. PHILLIPS

1220. **Derivative thermoanalytical techniques. Instrumentation and applications to thermogravimetry and differential thermal analysis.** C. Campbell, S. Gordon and C. L. Smith (Pyrotechnics Chem. Res. Lab., Picatinny Arsenal, Dover, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1188–1191.—The differentiator described consists of a battery-energised precision transmitting potentiometer coupled to the balancing slide-wire of the recorder used to obtain the primary thermogram. The signal generated by the transmitter circuit is differentiated by a resistance - capacity circuit and recorded on another time-base or x - y potentiometric recorder. The device has been used for recording curves that characterise a system with respect to its thermodynamic properties.

K. A. PROCTOR

Electrical

1221. **The rapidly dropping mercury electrode in direct-current and alternating-current polarography.** Nobuyuki Tanaka, Reita Tamamushi and Mutsuo Kodama (Tohoku Univ., Sendai, Japan). *Anal. Chim. Acta*, 1959, **20** (6), 573–580.—The characteristics of mercury electrodes with drop times <1 sec. in d.c. and a.c. polarography are investigated. These electrodes give attenuated maxima on the d.c. current - voltage curve. The maxima are suppressed by large amounts of polyoxyethylene lauryl ether and normal curves are obtained. In a.c. polarography the rapidly dropping electrode gives the peak current more accurately than the conventional electrode. H. N. S.

1222. **pH measurements with a glass electrode withstanding 1500 kg per sq. cm hydrostatic pressure.** A. Distèche (Lab. of Gen. Biology, Univ. of Liège,

Belgium). *Rev. Sci. Instrum.*, 1959, **30** (6), 474–478.—A layer of silicone oil separates the internal and external fluids and acts as a liquid piston to ensure pressure compensation. Such an electrode in conjunction with two silver - silver chloride electrodes maintains its hydrogen-electrode function at high pressure, and gives results for the pH changes resulting from dissociation-constant shifts caused by pressure that are in good agreement with the theoretically predicted values for HCl, acetic acid, carbonic acid and acetate buffer soln.

G. SKIRROW

1223. **Lead dioxide electrodes in acid - base titrations.** I. M. Issa and A. A. Abdul Azim (Chem. Dept., Cairo Univ.). *Egypt. J. Chem.*, 1959, **2** (1), 67–78 (in English).—The electrodes were prepared by electro-deposition on platinum from strongly acidic, weakly acidic and alkaline soln. All were unstable in soln. more alkaline than pH 3, but after being soaked for 3 hr. in 0·1 *M* NaOH the electrodes deposited from alkaline soln. became stable and gave reproducible titration curves. Examples are given of the titration of inorganic and organic acids with a precision better than 1%.

W. T. CARTER

1224. **Transistorised dead-stop end-point detector.** R. W. Freedman (5250 Keeport Drive, Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1287–1288.—The detector described is a more sensitive, transistorised version of the apparatus of Foulk and Bawden (*J. Amer. Chem. Soc.*, 1926, **48**, 2045). The unit is powered by a single battery; the electrode polarisation voltage can be varied over a wide range, and the variation of current with change of temp. can be compensated for rapidly.

K. A. PROCTOR

1225. **End-point location in controlled-potential coulometric analysis.** L. Meites (Dept. of Chem., Polytech. Inst. of Brooklyn, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (7), 1285–1286.—The modified technique described is simpler and more rapid than that previously described (*Ibid.*, 1955, **27**, 1116).

K. A. PROCTOR

1226. **Modified Sargent - Malmstadt automatic titrator for remote control use with plutonium solutions.** G. R. Waterbury (Univ. Calif., Los Alamos, N.M.). *Anal. Chem.*, 1959, **31** (7), 1138–1140.—The commercially available Sargent - Malmstadt instrument (*cf. Anal. Abstr.*, 1954, **1**, 3185) has been modified by replacing the gravity-flow burette with a motor-driven syringe, replacing the stirrer with a magnetic stirrer, re-wiring the titrator for remote operation and adding a micro-ammeter.

N. E.

1227. **Multi-purpose micro-separator (filtration, evaporation, centrifugation). Application to radioactivity measurements.** M. Herbain and D. Bertin (Serv. Rech. Roussel-Uclaf, Romainville, Seine, France). *Bull. Soc. Chim. Biol.*, 1959, **41** (4), 637–642.—The apparatus described consists of a glass jacket enclosed in a detachable metal mount, and has a multi-purpose lower end. For filtrations, this end is closed by a grid which supports the filtration medium, and is set in a holder connected to a vacuum source. For centrifuging, the grid and nozzle are replaced either by a flat-bottomed shutter, with or without a filter-paper, or by an independent cup for collecting the ppt. The latter arrangement may be used as a closed chamber for micro-evaporations in a controlled atmosphere. The separator measures 11 cm × 2·5 cm. For radioactivity measurements, this device is useful for producing thin, even layers of radioactive micro-precipitates, which can be counted directly.

D. B. PALMER

1228. **New proportional counters for gases and vapours.** R. Wolfgang and C. F. Mackay (Sterling Chem. Lab., Yale Univ., New Haven, Conn.). *Nucleonics*, 1958, **16** (3), 69–73.—Batch and flow-type counters into which the radioactive sample in gaseous form is introduced are described; such counters have a readily calculated efficiency of $\simeq 100\%$. The applications and use of proportional counters are compared with those of ionisation chambers and liquid scintillation systems.

G. J. HUNTER

1229. **Activation analysis with 14-MeV neutrons.** A. A. Lbov and I. I. Naumova. *Atomnaya Energiya*, 1959, **6** (4), 468–470.—Small amounts of O in metals and other materials were identified by the ^{16}N β-radiation produced in the reactions ^{16}O (n, p) ^{16}N (49 mb cross-section) during the irradiation of specimens by 14-MeV neutrons. The very short half-life of ^{16}N (7·3 sec.) prevented the radiochemical separation of N; however, the high energy of the β-particles (up to 10 MeV) was very effective in measuring the activity of the irradiated specimen. Experiments were also conducted for the determination of O in specimens by the reaction $^{16}O(t, n)$ ^{18}F; the results of both experiments were identical. The radioactivation analysis by 14-MeV neutrons was carried out with Si and P by the reaction $^{28}Si(n, p)^{28}Al$ with 220 mb cross-section and $^{31}P(n, \alpha)^{28}Al$ with 146 mb cross-section. The method was also used for the determination of S and Cl^- in organic materials, *e.g.*, methacrylates, and for the determination of P, S and Cl^- in graphite.

NUCL. SCI. ABSTR.

1230. **Mass spectrometer for chemical use.** E. Schumacher (Chem. Inst., Zürich Univ.). *Helv. Chim. Acta*, 1959, **42** (4), 1248–1257.—The construction and operation are described of a first-order direction-focusing mass spectrometer, with a homogeneous 60°-sector magnet field of 20 cm radius. The instrument is suitable for isotopic and quant. analyses by means of surface ionisation, for precise measurements of small isotope effects in gases and vapours with a double feed and interceptor system, and for isotope frequency and fragmentation studies on gases by means of electron recoil and field ionisation. The isotopic analysis of RbCl is described; this gives the ratio of ^{87}Rb to ^{85}Rb as $0{\cdot}3860 \pm 0{\cdot}0009$.

J. L. PROSSER

1231. **Application to mass spectrometry of automatic manometer for batchwise gas sampling.** J. Malli, G. Wood and R. A. Friedel (Bureau of Mines, U.S. Dept. of the Interior, Pittsburgh, Pa.). *Anal. Chem.*, 1959, **31** (7), 1283.—The advantages of using an automatic manometer are that uniform pressures of samples allow easier checking and comparing of results; and that the analytical accuracy is increased somewhat by the high precision of measurement of the calibration and sample pressures. The unit described has not been used at pressures >120 torr.

K. A. PROCTOR

1232. **New method for the determination of water content in tobacco.** V. Dukat (Res. Inst. Tobacco Ind., Báb, Czechoslovakia). *Průmysl Potravin*, 1959, **10** (6), 290–291.—An apparatus based on the measurement of dielectric constant is described which enables 15 to 20% of H_2O in tobacco to be determined with an accuracy within $\pm 0{\cdot}4\%$. A constant pressure of the electrodes on the measured material must be applied.

J. ZÝKA

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{3+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

Volume 7. No. 3, Abstracts 848–1232 March, 1960

ANALYTICAL ABSTRACTS

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Printed and published for the Society for Analytical Chemistry by W. Heffer & Sons Ltd., Cambridge, England.
Communications to be addressed to the Editor, Norman Evers, 14, Belgrave Square, London, S.W.1.
Enquiries about advertisements should be addressed to Walter Judd Ltd., 47, Gresham Street, London, E.C.2.
Entered as Second Class at New York, U.S.A., Post Office.